Approaches to Photo-activated Cytotoxins

A thesis
submitted in partial fulfillment
of the requirements for the degree
of
Doctor of Philosophy in Chemistry
at the University of Canterbury
by

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University of Canterbury
Christchurch
New Zealand
2006

Acknowledgements

Reflecting of nearly four years of study in such an amazing place (New Zealand), a long list of people to whom I'm indebted for their contributions to this work soon emerges. Since a long list of acknowledgements may not be sensibly included here, the following highly condensed version must suffice.

First and foremost, I would like to acknowledge my supervisor, Associate Professor Richard Hartshorn, for his help, guidance, and supervision throughout the duration of this work. Richard's dedication as a teacher and mentor has been outstanding. His enthusiasm for chemistry and generosity of spirit can not be thanked enough.

I'm also extremely grateful to have benefited from Prof. Ward Robinson's great knowledge and his passion for teaching X-ray crystallography. Thanks Ward for the amount of time you spent with me in the X-ray lab.

I would also like to thank many of the academic staff in the Chemistry Department for their "open-door" policy, in particular Prof. Peter Steel (X-ray), Dr. Jan Wakira (X-ray), Prof. John Blunt (NMR), Associate Prof. Bryce Williamson (HoD), Dr. Owen Curnow, and Dr. Alison Downard for their time and their expertise.

My special thanks also go to Dr. Carl Otter for helping me in the lab during the first year of my research, to William Lewis for helping me in X-ray crystallography, and also to Dr. Sean Devenish for rescuing me on many occasions while running NMR experiments.

I also wish to acknowledge all those who have passed through Lab. 831 during the past four years, I have enjoyed your company very much. The Hartshorn group lab., and the department as a whole, has been a friendly and enjoyable environment in which to study and learn a deep kiwi culture. It has been a great pleasure to share political, cultural, scientific, historical, and religious views with Julian Browne, Prof. Jim Coxon, Dr. Thomas Francis, Dr. Colin Freeman, Martin Lee, and Dr. Andy Pratt. Thank you all for being interested in my Persian culture.

I would also like to thank Prof. Andrew Abell and his research students for letting me use their distillation apparatus in their lab.

My thanks also to the technical staff in the department who work diligently away, often unseen. Particular thanks to Bruce Clark for running of many mass spectrometric analysis, to Wayne Mackay for maintaining my pumps and electrical devices, to Rewi Thompson for his guidance in the NMR lab, to Sandy Ferguson and Russell Gillard for maintaining the diffractometer, and to Robert McGregor for fixing my glassware.

I would also like to express my thanks to the department administrators, Rachael Stokes, Diane Caddick, and more recently to Noelene Longley for their excellent office work during my research.

I wish to extend special thanks to the technical staff in the store, particularly to Bruce Whitfield for the many ways in which he has contributed to my Ph.D. – as a source of patience, encouragement – and as a friend.

The assistance of a University of Canterbury Doctoral Scholarship is also gratefully acknowledged.

My family members (specially Maman, Baba, my brother, Hamid, and his wife, Ulrike) have supported me throughout my studies and I can never thank them enough for their love, support, and encouragement.

Special thanks must go to my partner, Michelle, who allowed me to put things into perspective on many occasions; her endless loving, caring, friendship, leadership, and understanding helped me where I am today. Michelle, thanks for teaching me English and the Kiwi life style.

I would also like to thank my friend, David, his wife, Monica, and their son, Daniel (Ti-Ti) in Singapore for their huge support, kindness, and encouragement. Without them, this work would have been impossible, as I wouldn't be able to come to New Zealand at all. You will remain the symbol of humanity and friendship in my life.

To Those Suffering With Cancer!

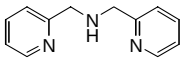
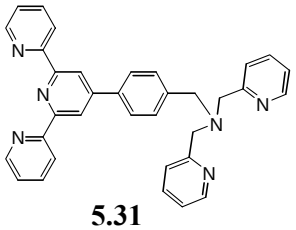
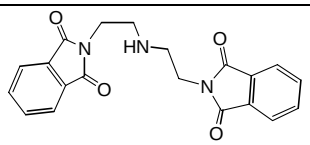
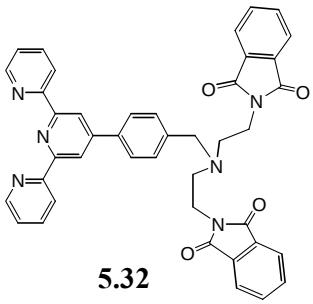
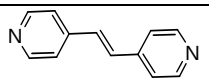
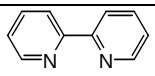
Table of Contents

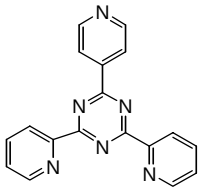
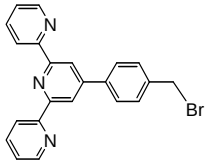
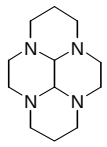
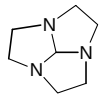
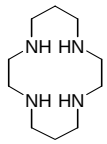
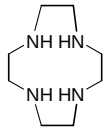
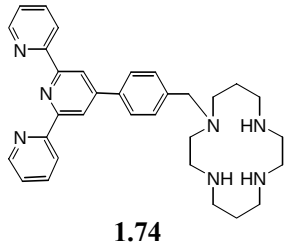
Abbreviations

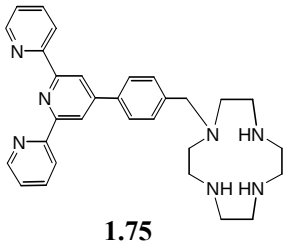
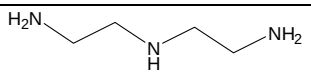
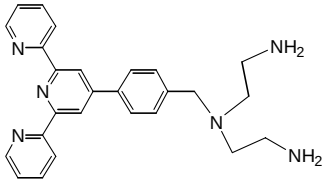
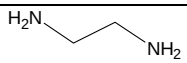
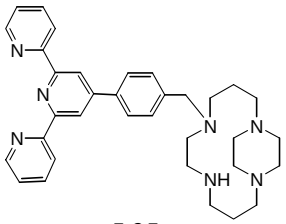
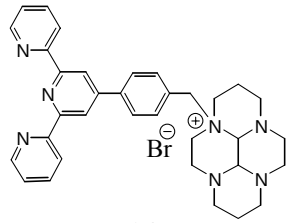
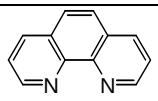
A	acceptor
BPO	benzoyl peroxide
C	Cytosine
conc	concentrated
COSY	2D ^{1}H NMR correlation spectroscopy
D	donor
DMF	*N,N*-dimethylformamide
DMN	dimethoxynaphthalene
DMSO	dimethylsulfoxide
DNA	deoxyribonucleic acid
ESI-MS	electrospray ionisation mass spectrometry
fac	facial
G	Guanine
GS	ground state
HSC	hypoxia-selective-cytotoxin
IC	internal conversion
IR	infra-red frequency light
ISC	intersystem crossing
LHD	light-harvesting device
M	moles per litre
^{3}MC	dd metal-centred triplet state

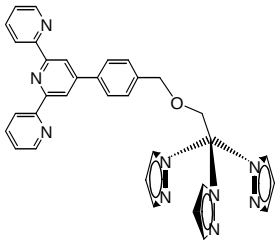
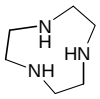
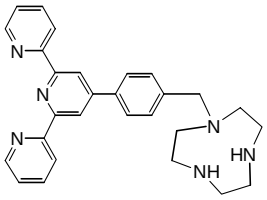
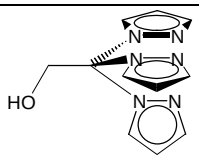
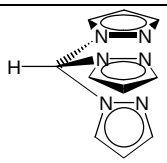
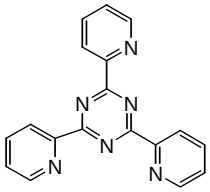
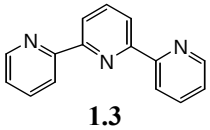
L	linker
LMCT	ligand-to-metal-charge-transfer
mer	meridional
MLCT	metal-to-ligand-charge-transfer
^{1}MLCT	metal-to-ligand-charge-transfer singlet state
^{3}MLCT	metal-to-ligand-charge-transfer triplet state
NBS	*N*-bromosuccinimide
nm	nanometre
NMR	nuclear magnetic resonance
NOESY	nuclear Overhauser effect spectroscopy
PDT	photodynamic therapy
ppm	parts per million
R_1	refinement factor
RNA	ribonucleic acid
r.t	room temperature
T	Thymine
THF	tetrahydrofuran
TMS	tetramethylsilane
TPMS	3-(trimethylsilyl)-1-propane sulfonic acid
Ts	tosylate
U	Uracil
UV	ultra-violet frequency light
vis	visible light
λ_{max}	wavelength at maximum absorbance
ε_{max}	extinction coefficient at maximum absorbance

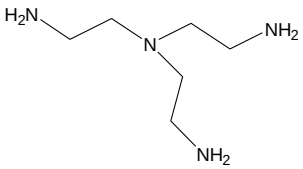
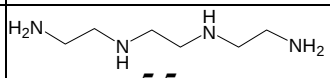
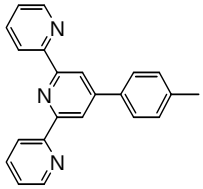
Abbreviations for Ligands

Abbreviation	Name of Ligand	Chemical Structure and Number
bpa	bis(2-picolyl)amine or *N,N′*-bis(2-pyridylmethyl)amine	
bpat	4'-(*p*-(1,5-bis(phthalimido)-3-azapentan-3-yl)methylphenyl)-2,2':6',2''-terpyridine	 **5.31**
bpta	1,5-bis(phthalimido)-3-azapentane	 **5.7**
bptt	4'-(*p*-(1,5-bis(phthalimido)-3-azapentan-3-yl)methylphenyl)-2,2':6',2''-terpyridine	 **5.32**
bpe	1,2-bis-(4-pyridyl)-ethylene	
bpy	2,2'-bipyridine	 **1.1**

bppt	2,4-bis(2'-pyridyl)-6(4"-pyridyl)-1,3,5-triazine	 **2.1**
btp	4'-(*p*-bromomethylphenyl)-2,2':6',2"-terpyridine	 **1.50**
bisaminal-cyclam	*cis*-decahydro-1*H*,6*H*-3a,5a,8a,10a-tetraazapyrene	 **5.20**
capped-tacn	1,4,7-triazatricyclodecane	 **5.17**
cyclam	1,4,8,11-tetraazacyclotetradecane	 **5.1**
cyclen	1,4,7,10-tetraazacyclododecane	 **5.2**
cymt	4'-(*p*-(1,4,8,11-tetraazacyclotetradec-1-yl)methylphenyl)-2,2':6',2"-terpyridine	 **1.74**

cynt	4'-(*p*-(1,4,7,10-tetraazacyclododec-1-yl)methylphenyl)-2,2':6',2"-terpyridine	 **1.75**
dien	diethylenetriamine	 **5.4**
dint	4'-(*p*-(1,5-bis(amino)-3-azapentan-3-yl)methylphenyl)-2,2':6',2"-terpyridine	 **5.33**
en	1,2-diaminoethane	
pcymt	4'-(*p*-(1,5,8,12-tetraazabicyclo[10.2.2]hexadec-5-yl-methylphenyl)-2,2':6',2"-terpyridine	 **5.35**
ptmtb	4'-(*p*-(10bα,10cα)-decahydro-3a-1*H*,6*H*-3a,5a,8a,10a-tetraazapyrenium-methylphenyl)-2,2':6',2"-terpyridine bromide	 **5.34**
phen	1,10-phenanthroline	 **1.2**

pzt	4'-(*p*-(2,2,2-tris(1*H*-pyrazol-1-yl)ethoxymethylphenyl)-2,2':6',2"-terpyridine	 **1.71**
tacn	1,4,7-triazacyclononane	 **5.3**
tcnt	4′-(*p*-(1,4,7-triazacyclonon-1-yl)methylphenyl)-2,2′:6′-2″-terpyridine	 **5.8**
tpe	2,2,2-tris(1*H*-pyrazol-1-yl)ethanol	 **4.1**
tpm	tris(1*H*-pyrazol-1-yl)methane	 **3.1**
tpt	2,4,6-tris(2-pyridyl)-1,3,5-triazine	 **1.70**
tpy	2,2':6',2"-terpyridine	 **1.3**

tren	tris(2-aminoethylamine)	
trien	triethylenetetraamine	 **5.5**
ttp	4'-(*p*-tolyl)-2,2':6',2"-terpyridine	 **1.48**

Abstract

The synthesis and coordination chemistry of eleven bridging ligands, eight of which are new compounds, are described. These ligands are all based on the tridentate terpyridyl system. The other metal ion binding sites of these ligands contain pyridine/bipyridine/pyrazole rings or amine/azamacrocycles domains. In these ligands, the two metal ion binding sites are differentiated by the number of atoms in each site, the configuration of the binding site or the types of donor atom that are present. This binding site differentiation allows to use the different coordination properties of the binding sites to control the regiochemistry of the complexation, ensuring that the correct metal ion is incorporated at the correct binding site in the ligand.

Many of the complexes synthesised are mono-ruthenium(II) complexes where Ru(II) ions are situated in the terpyridyl sites of the ligands. These include heteroleptic Ru(II) complexes of the type $[Ru(ttp)(L)]^{2+}$, where ttp is 4'-(*p*-tolyl)-2,2':6',2''-terpyridine, and L is the bridging ligand.

Reactions of the Ru(II) complexes with a range of metal ions including Co(III) ion have been investigated. The Ru(II) complexes can be classified into three main categories depending on the type of ligands that have been employed: (1) Ru(II) complexes which can react with Co(III) ions to form heterodinuclear Ru(II)-Co(III) complexes; (2) Ru(II) complexes which react only with Ag(I) ions and no other common metal ions that we have tried; (3) Ru(II) complexes with no detectable ability to coordinate other common metal ions.

Following standard cobalt chemistry, some heterodinuclear Ru(II)-Co(III) complexes of the type $[(ttp)Ru(cymt)Co(X)_2]^{3+}$, where X = NO_2^-, Cl^-, and OH^-, have

been successfully prepared from the corresponding Ru(II) complexes. In these heterodinuclear complexes, anions such as NO_2^-, Cl^-, or OH^- can be readily attached to the Co(III) ions. However, attachment of a neutral species such as en ligands to the Co(III) ions in the complexes proved to be more difficult. Reactions of heterodinuclear Ru(II)-Co(III) complexes with en ligands result in removal of the cobalt ions from the complexes. This is may be a result of a significant difference in the overall charges between the complexes with anionic and the complexes with neutral ligands (3+ *vs* 5+). Higher overall charge of the complexes when protonable ligands such as monodentate en are present, may destabilize the complexes even more.

A combination of NMR spectroscopy, ESI-MS, UV-vis spectroscopy, elemental analysis, and X-ray crystallography has been used to characterise the ligands and their complexes. The crystal structures of one new ligand and sixteen complexes are described.

Chapter 1

Introduction

1.1 Introduction

A major problem in the development of cytotoxic anti-cancer drugs is to achieve discrimination between cancer cells and normal tissue. Often, the more rapid proliferation of cancer cells compared with (most) normal cells is targeted. As a result, most drugs in use are anti-proliferative agents which target DNA, either directly (*e.g.* alkylating agents) or *via* its ancillary enzymes (*e.g.* topoisomerase inhibitors). However, poor discrimination between cancer cells and other rapidly dividing cells in the body can still lead to the well known debilitating side effects of chemotherapy.

Another problem often encountered in cancer therapy is the presence of tumour cell subpopulations that are resistant to treatment. Solid tumours frequently contain hypoxic cells that are resistant to killing by ionizing radiation and also by many chemotherapeutic agents. However, these hypoxic cells can be exploited for therapy by non-toxic hypoxic-activated prodrugs. Bioreductive drugs require metabolic reduction to generate cytotoxic metabolites. This process is facilitated by appropriate reductases and the lower oxygen conditions present in solid tumours. The unique presence of hypoxic cells in human tumours provides an important target for selective cancer therapy.

In order to understand the research project I have undertaken, it is essential to gather a wide range of material in the area of donor-accepter systems and their applications in the area of photo-activated cancer chemotherapy.

Inorganic and coordination complexes have a long history of use as chemotherapeutic agents. The successful application of inorganic complexes as drugs involves the recognition of their bioinorganic modes of action coupled with the traditional pharmacokinetic parameters of uptake, distribution and excretion.[1] Further, the rational approaches to chemotherapy and particularly the notion of selective toxicity[2] must be placed in an inorganic context. The concept of selective toxicity, and its scientific basis, is of particular use in describing the actions of drugs on an invading organism, be it viral, bacterial, parasitic, or ultimately malignant tumours caused by the cancerous growth of the host's cells. The achievement of some form of selectivity is critical to the successful use of any agent as a drug or modifier of biological response.[1]

This research project is aimed at developing inorganic molecules containing Ru(II) and Co(III) ions which will release and activate a cytotoxin when they are hit by light of a particular wavelength. The use of visible (preferably red) light, which does not damage tissue, would stand in marked contrast to current approaches to enhance the efficiency the anti-tumour agents, many of which utilise X-ray photons (*e.g.* oxygen mimetic radiosensitisers[3]).

The beginning of this chapter outlines the reports on the recent studies by other groups in this area of chemistry.

Methods of constructing of bridging ligands for my purpose are described later in this chapter.

1.2. Chemotherapy

On present statistics, one in three New Zealanders will develop cancer in their lifetime.[4] Overall cancer incidence rose by almost 21 percent in the five years between 1991 and 1995. The New Zealand cancer mortality rate of 142.8 per 100,000 population ranked 20th highest out of 22 developed countries.[4]

Population growth and rising cancer incidence dictate an increasing demand for cancer therapies. Given the inherent tendency of all cancers to metastasise, chemotherapy will remain a major component of cancer treatment. It is also important in cases where a tumour is inoperable. Major advances in the cure of cancer are likely to come from improvements in systemic treatment.

The treatment of cancer over the past 50 years has relied on three main approaches: surgery, radiotherapy, the use of anticancer medicines (chemotherapy) or a combination of them.

The term chemotherapy, as defined by Paul Ehrlich (the undisputed 'father' of chemotherapy) means the use of drugs to injure an invading organism without damage to the host.[1] Historical accounts of the development of chemotherapy are to be found in Albert's Selective Toxicity[2] and in The Molecular Basis of Antibiotic Action.[5] The utility of any substance as a chemotherapeutic agent will clearly depend on how its action favours the host over the invading organism or 'uneconomic species'. By selective attack, the drug can augment the host's natural defence, which will be responsible, in the last resort, for the elimination of unwanted species.

Chemotherapy uses toxicity in the service of man and requires an essentially irreversible process (cell death). Normally a different organism is attacked – virus, bacterium, parasite – but in cancer the host's own malignant cells are attacked. The

achievement of selectivity in this latter case is clearly a matter of great difficulty.[1] This will be discussed later in this chapter.

1.2.1. Interaction of Metal Complexes with DNA

A basic aim of chemotherapy is clearly to obtain a selective effect between a normal host cell and other cells, be they bacterial, protozoal, or cancerous. This selectivity may be achieved by differences in distribution, comparative biochemistry, or comparative cytology,[2] the damage induced eventually renders the attacked cell more susceptible to the host's defence mechanisms.

The predominant role of DNA in cellular replication and transmission of genetic information makes the nucleic acids a primary target for drug action, and many drugs are considered to act fundamentally at this level.[5] The inhibition of synthesis of DNA may be direct, involving binding and/or subsequent damage such as strand breakage to the target, or indirect, employing an interface with the synthesis of critical precursors necessary for DNA synthesis.[6]

The studies reviewed by Farrell[1] show that metal complexes act by both mechanisms.

Figure 1.1 shows the basic sites of interaction and possible modes of interaction with DNA, as exemplified by common drugs and also metal complexes. The interactions may be summarised as:

1. Intercalation
2. Outer-sphere binding
3. Inner-sphere (groove) binding
4. Strand breakage

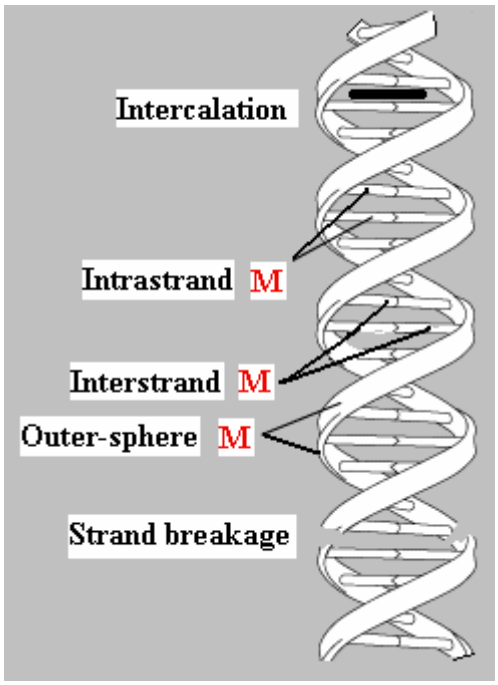


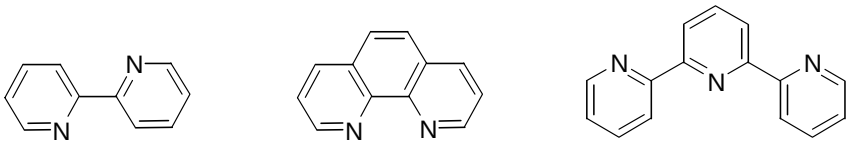
Figure 1.1. Schematic presentation of the modes of interaction of metal complexes with DNA. (M is a metal ion such as Pt(II)).

1.2.1.1. Intercalation

Intercalation involves the insertion of a planar molecule between two neighbouring DNA base pairs, to which it is held by Van der Waals forces.[1] A further weak interaction of an electrostatic type between the phosphate anions and charged groups of the molecule may also be present. The classical view of intercalation requires that the helix be extended and locally unwound by the binding interaction.[7-8,9] Some classes of intercalators are mentioned below:

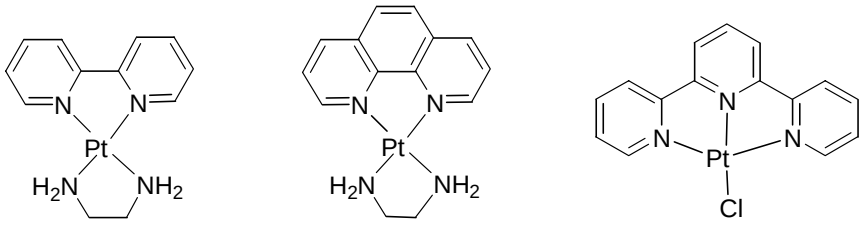
1.2.1.1.1. Platinum Complexes

The intercalation of platinum complexes containing bidentate or tridentate aromatic ligands (Figure 1.2) was proposed for binding to tRNA and then to DNA.[7] The structures of the relevant complexes of platinum are shown in Figure 1.3. The prerequisite for binding is the presence of planar ligands such as 2,2'-bipyridine (bpy), **1.1**, *o*-phenanthroline, **1.2**, 2,2':6',2"-terpyridine (tpy), **1.3**, which are in the plane of the square planar complexes formed by the four ligand donor atoms and the metal ion.



1.1 **1.2** **1.3**

Figure.1.2. Bidentate and tridentate pyridine based ligands.



1.4 **1.5** **1.6**

Figure 1.3. Structures of some platinum complexes containing planar aromatic ligands that have been studied as intercalators.

1.2.1.1.2. Complexes Containing 1,10-Phenanthroline Ligands

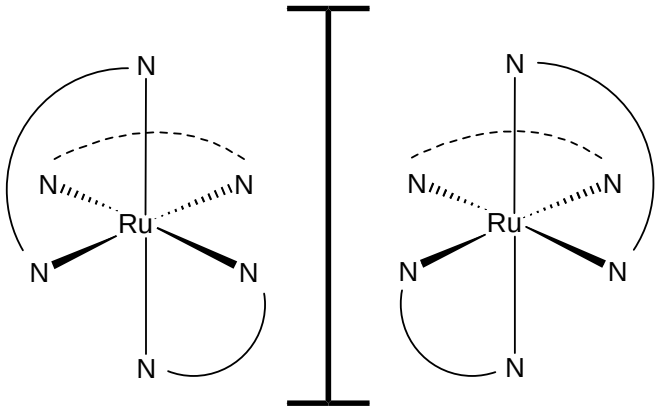
The chelating ligand **1.2** is capable of sufficient overlaps with base pairs, and their use of metal complexes as intercalating agents and probes of DNA structure is

being explored in a series of interesting studies.[8,9] In this case coplanarity is not always possible as the geometry varies from octahedral [$M(phen)_3$] to tetrahedral or square planar [$M(phen)_2$]. The phenanthroline complexes have been studied extensively for their biological activity.[10]

Studies on the Zn complexes, [$ZnCl_2(phen)$], [$Zn(phen)_2]^{2+}$, and the octahedral [$Zn(phen)_3]^{2+}$, showed that all the complexes unwound closed circular DNA, indicative of intercalation.[11]

Octahedral tris(phenanthroline) complexes of Ru(II), Co(III), and Rh(III) are substitutionally inert, enantiomeric (Figure 1.4) and the isomers were shown to bind to the double helix.[11,12]

One of the most well-studied members in this group is tris(phenanthroline)ruthenium(II) ([$Ru(phen)_3]^{2+}$). Many experimental techniques have been applied to study the interaction of this compound with DNA, but despite this its binding mode and its effect on the DNA structure are uncertain and have been the subject of much controversy.[13-15] Unwinding studies with closed circular DNA and absorption and fluorescence spectroscopy data were proposed as evidence that both the Δ and the Λ enantiomers of [$Ru(phen)_3]^{2+}$ bind to DNA by intercalation.[15-17] From 1D NMR it was first inferred that Δ intercalates and Λ binds in the minor groove[18] while 2D NMR later indicated minor groove contacts, but no proper intercalation, for both enantiomers.[15] From linear dichroism, equilibrium, and viscosity experiments it had been concluded that neither Δ nor Λ is bound to DNA by intercalation.[19] Relatively recent studies showed the Δ and Λ enantiomers of [$Ru(phen)_3]^{2+}$, bind reversibly and with low affinity to DNA.[15]



Λ-$[Ru(phen)_3]^{2+}$ Δ-$[Ru(phen)_3]^{2+}$

Figure. 1.4. Optical isomers of complexes of phenanthroline, $[Ru(phen)_3]^{2+}$.

1.2.1.1.3. Rhodium(III) Complexes

The 1.2 Å resolution crystal structure of a sequence-specific octahedral rhodium intercalator Δ-α-$[Rh[(R,R)\text{-}Me_2trien]phi]^{3+}$ (where (R,R)-Me_2trien is 2*R*,9*R*-diamino-4,7-diazadecane and phi is 9,10-phenanthrenequinone diimine) bound to a DNA helix provides a rationale for the sequence specificity of rhodium intercalators.[20] It also explains how intercalation in the centre of an oligonucleotide modifies DNA conformation. Barton *et al*.[20] also indicate that the rhodium complex intercalates *via* the major groove where specific contacts are formed with the edges of the bases at the target site. The ligand is deeply inserted into the DNA base pair stack. The primary conformational change of the DNA is a doubling of the rise per residue, with no change in sugar pucker from B-form DNA. Based upon the five crystallographically independent views of an intercalated DNA helix observed in this structure, the intercalator may be considered as an additional base pair with specific functional groups positioned in the major groove.[20]

1.2.1.1.4. Porphyrins and Metalloporphyrins

A further series of complexes that has been suggested to intercalate is based on tetrakis(4-*N*-methylpyridiniumyl)porphyrin (Figure 1.5).

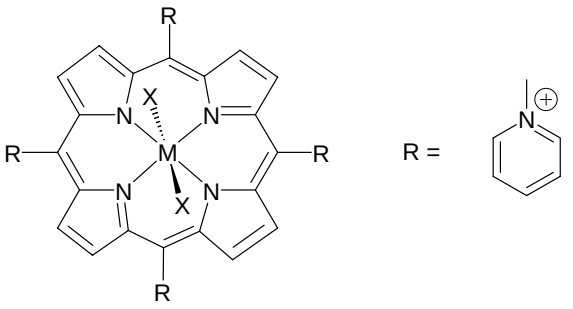


Figure. 1.5. Tetrakis(4-*N*-methylpyridiniumyl)porphyrin complexes. (M = Co(III), Mn(III), Fe(III), Ni(II), Zn(II), or Pd(II)). The axial substituent X may be present or absent depending on the metal M.[21]

The intercalation of metalloporphyrin systems has been examined by a wide range of techniques and in fact presents a good example of how varying techniques can give information on modes of DNA binding. These methods include NMR, CD, fluorescence and resonance Raman as well as physical studies such as viscosity and melting.[21,22] The intercalation of the free porphyrin with DNA is judged to occur on the basis of studies on the effects on the UV spectrum, viscosity changes and interaction with closed circular DNA.[1] Extension to metal complexes gave the interesting resul that whereas the copper complex interacted with poly(dG-dC), complexes with Ni(II), Co(III), Zn(II), Fe(III), and Mn(III) did not react; however, all these reacted with poly(dA-dT) and also with DNA in a nonintercalative, presumably electrostatic manner. The presence of the axial ligands for Zn, Co, Fe, and Mn is considered to inhibit the intercalation sterically. The copper complex, with no axial ligands, behaves like the free porphyrin.

1.2.1.2. Outer Sphere Binding

The negatively charged backbone of the DNA helix may interact with a variety of positively charged molecules such that, either through coulombic interactions or phosphate-oxygen binding, the overall charge is reduced with subsequent effects on stability and conformation.

A further set of complexes that has been shown to interact with DNA by outer sphere binding is of the inert metal-amine type. The most studied system is that of $[Co(NH_3)_6]^{3+}$ and its ethane-1,2-diamine analogue. The inert nature of these species indicates reaction by an outer sphere mechanism and the main interaction is considered to be coulombic attraction. Hydrogen bonding either directly to phosphate or *via* water molecules can also contribute to the overall stabilization.[23]

1.2.1.3. Inner Sphere (Covalent) Binding

A number of aqua metal ions bind covalently to the bases of DNA. Early examples of this mode of binding were mercury and silver.[1] In the case of silver, the metal crosslinks stabilize the helix.[24,25]

Crosslinking with Zn(II) and Cu(II) has also been proposed to explain their DNA binding.[26]

The above examples relate to simple metal ions without a coordination environment complicated by ligands other than by water molecules. As for outer sphere binding, ligand substitution alters the affinity and specificity of the binding interaction.[1]

One of the best defined series is that of the mixed platinum-chloroamines, from $PtCl_4^{2-}$ to $[Pt(NH_3)_4]^{2+}$. As shown in Figure 1.6, for example, the platinum centre in the

structure of *cis*-[Pt(NH_3)$_2$(d(pGpG))][27] coordinates to the N7 position of the guanine bases. In terms of interactions with the full polynucleotide, it is likely that the *cis*-diamineplatinum centre, with two coordination sites available, would yield an intrastrand crosslink between neighbouring guanine residues on a strand.[28]

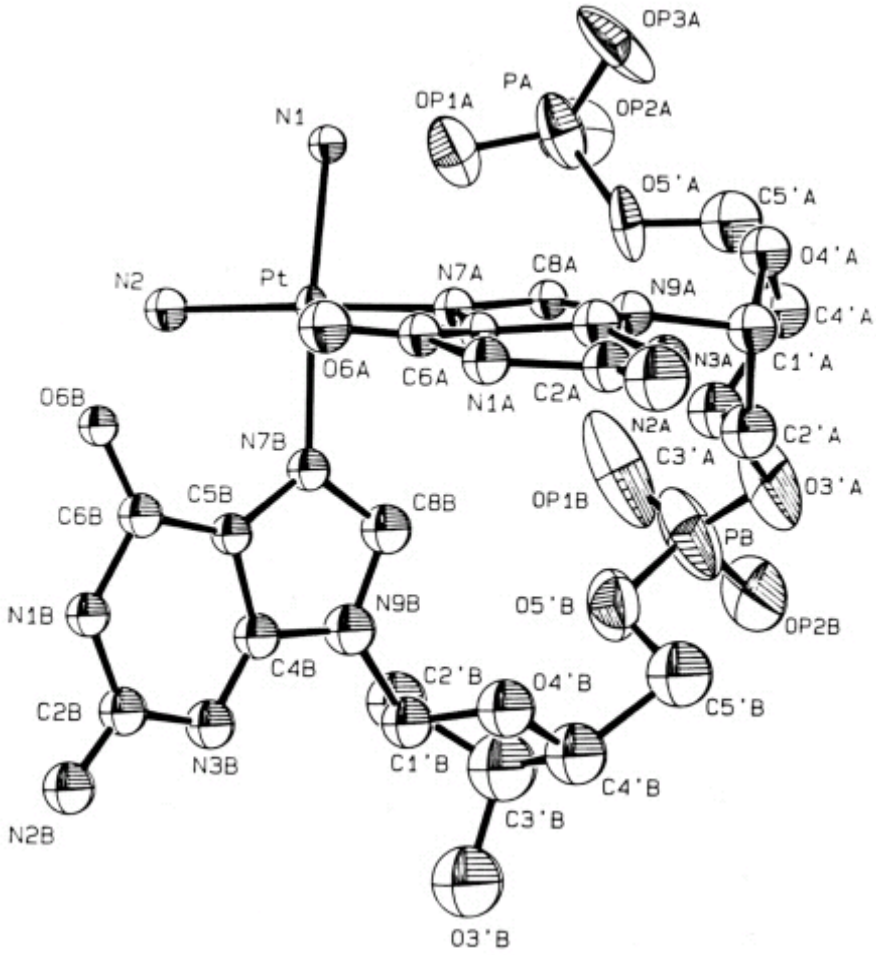
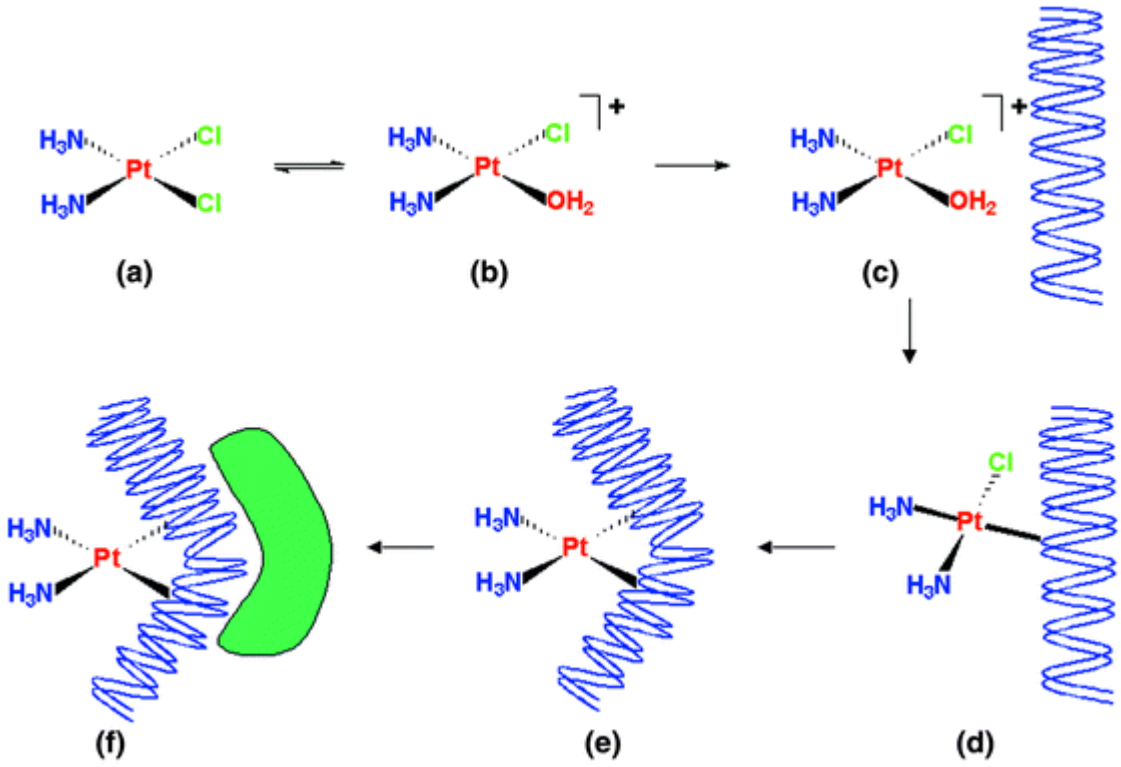


Figure 1.6. Molecular structure of *cis*-[Pt(NH_3)$_2$(d(pGpG))].[28,29]

Other nucleophilic sites targeted by soft metal ions on the bases include the N7 position of adenine, the N3 position on cytosine, and the deprotonated N3 position on thymine and uracil. Some additional covalent binding to the N1 position of the purines has also been observed.

The interaction of cisplatin with DNA is a multistage process as shown in Scheme 1.1, and involves at least: aquation, preassociation, monofunctional adduct

formation, closure to a bifunctional adduct perhaps preceded by aquation, distortion of DNA and recognition of this distortion by a variety of proteins.[30]



Scheme 1.1. The sequence of events in cisplatin binding to DNA.[30] **(a)** and **(b)** aquation; **(c)** preassociation; **(d)** monofunctional adduct formation; **(e)** closure to a bifunctional adduct; **(f)** recognition of the DNA distortion by a variety of proteins.

Some structural consequences of platinum binding to DNA have been recently discussed by Hambley.[30] He also described the understanding of the factors that control the various stages of platinum binding to DNA shown in Scheme 1.1. Information on the consequences of platinum binding to DNA in terms of bending and unwinding has been available from electrophoresis studies by others,[31,32] but it is only relatively recently that structural details have begun to emerge from crystallographic and NMR analyses.

A number of different adducts are formed that evidently contribute differently to the anticancer effect and side effects.[30] Platinum binding to DNA ultimately leads to cell death. An important aspect of this process is believed to be high-mobility group (HMG) domain protein recognition of and binding to DNA bent by the platinum adduct.[30] HMG domain protein binding is believed to interfere with the repair and removal of the platinated site by the normal repair mechanisms and this interference probably initiates the cell death process.[30]

1.2.1.4. Strand Breakage

Strand breakage, either single strand or double strand, involves covalent bond cleavage of the helix.[33] In principle, the strand breaking reactions of transition metal complexes with polynucleotides generally fall into two categories: (i) those involving a redox reaction of the metal complex that mediates oxidation of the nucleic acid; and (ii) those involving coordination of the metal centre to the sugar-phosphate backbone so as to mediate hydrolysis of the polymer. Both redox and hydrolytic reactions of the metal complexes with nucleic acids have been exploited with much success in the development of tools for molecular biology.

The simplest redox reaction with polynucleotides one might consider as an illustration is the Fenton reaction, which indirectly promotes DNA strand breakage through radical reaction on the sugar ring.[1,28] The reaction with $[Fe(EDTA)]^{2-}$ is shown in Figure 1.7.

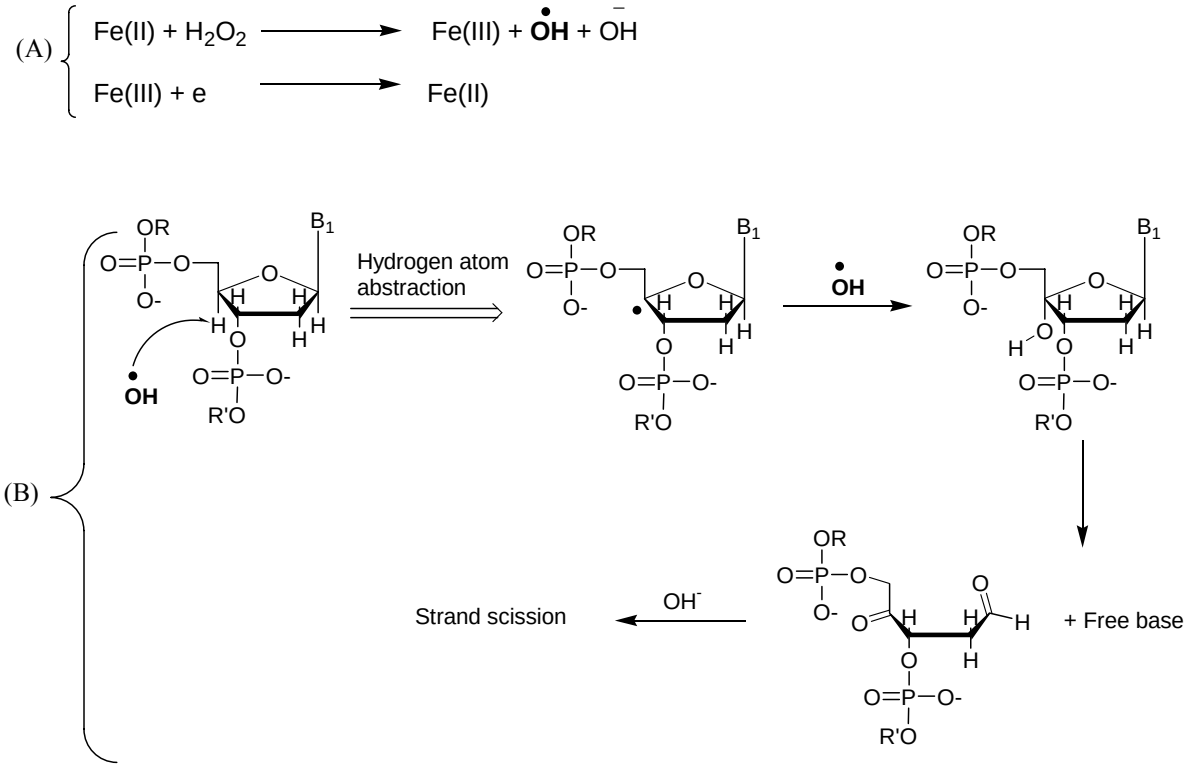


Figure 1.7. An illustration of DNA strand cleavage mediated by hydroxyl radicals produced by the Fenton reaction (A) of $[Fe(EDTA)]^{2-}$ with hydrogen peroxide. The cleavage scheme (B) shows the products obtained as a result of initial C4'-H abstraction by the hydroxyl radicals.

Ferrous ion, in the presence of hydrogen peroxide, generates hydroxyl radicals, and in presence of a reductant such as mercaptoethanol, the hydroxyl radical production can be made catalytic. Although ferrous ion itself does not appear to interact appreciably with a nucleic acid, especially when chelated in an anionic EDTA complex and repelled by the nucleic acid polymer, the radicals, produced in appreciable quantities catalytically, attack different sites on the sugar ring, indirectly yielding breakage of the sugar-phosphate backbone.

Hydrolysis reactions of the phosphodiester linkage of polynucleotides appear preferable to redox-mediated cleavage reactions, since in the hydrolytic reaction all information is preserved. In redox cleavage by sugar oxidation, for example, both a sugar fragment and free nucleic acid base are released from the polymer, and, in contrast to hydrolytic chemistry, the direct relegation of the fragments becomes practically impossible.

Metal ions can be effective in promoting hydrolysis of the phosphodiester, since they can function as Lewis acids, polarising the phosphorous-oxygen bond to facilitate bond breakage, and can also deliver the coordinated nucleophile to form the pentacoordinate phosphate intermediate. Figure 1.8 illustrates one crystallographically characterised model system developed by Sargeson and co-workers,[34] where hydrolysis of a model phosphodiester was enhanced dramatically by taking advantage of both the acidic and the nucleophilic characteristics of the bound Co(III) species.

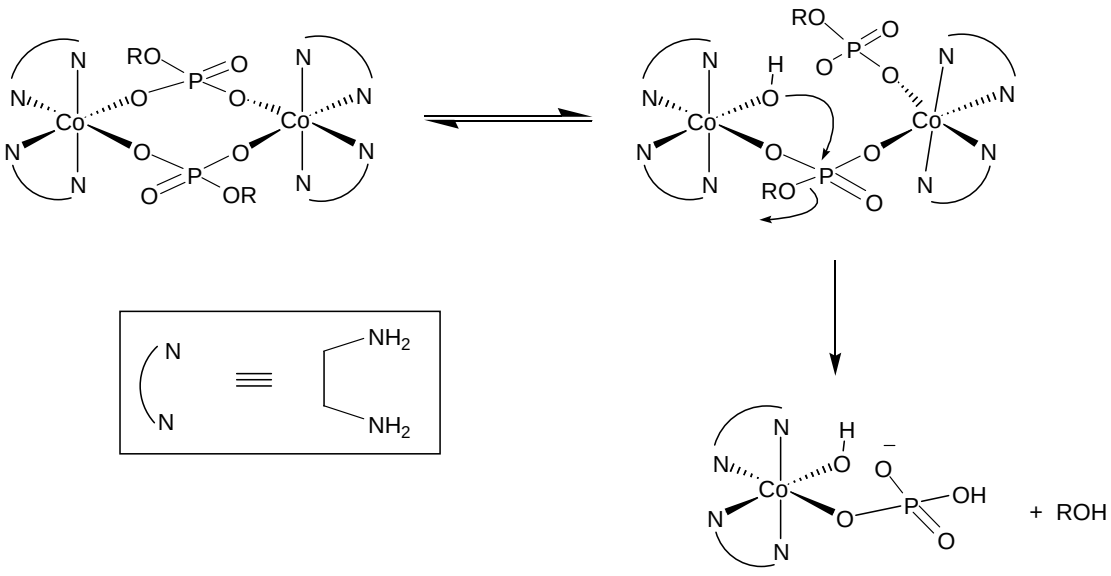


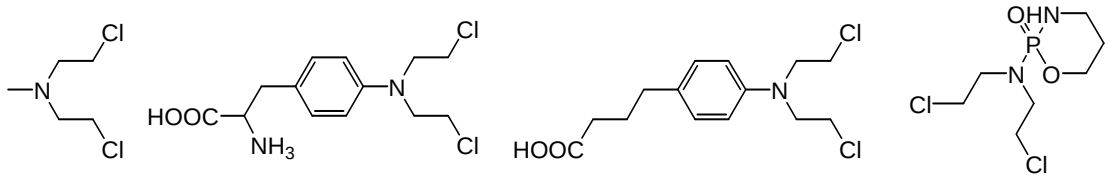
Figure 1.8. Illustration of a phosphate ester hydrolysis in a dinuclear model complex catalysed by coordinated cobaltic ions, with one metal ion functioning as a Lewis acid and the other functioning to deliver the coordinated hydroxide.[34]

1.2.2. DNA Alkylating Agents

In 1962, Ross[35] defined a biological alkylating agent as a compound which can replace a hydrogen atom by an alkyl group under physiological conditions (pH 7.0 – 7.4, 37 ºC, aqueous solution). Alkylating agents were the first group of chemicals systematically investigated in order to find effective inhibitors against neoplastic cells.[36]

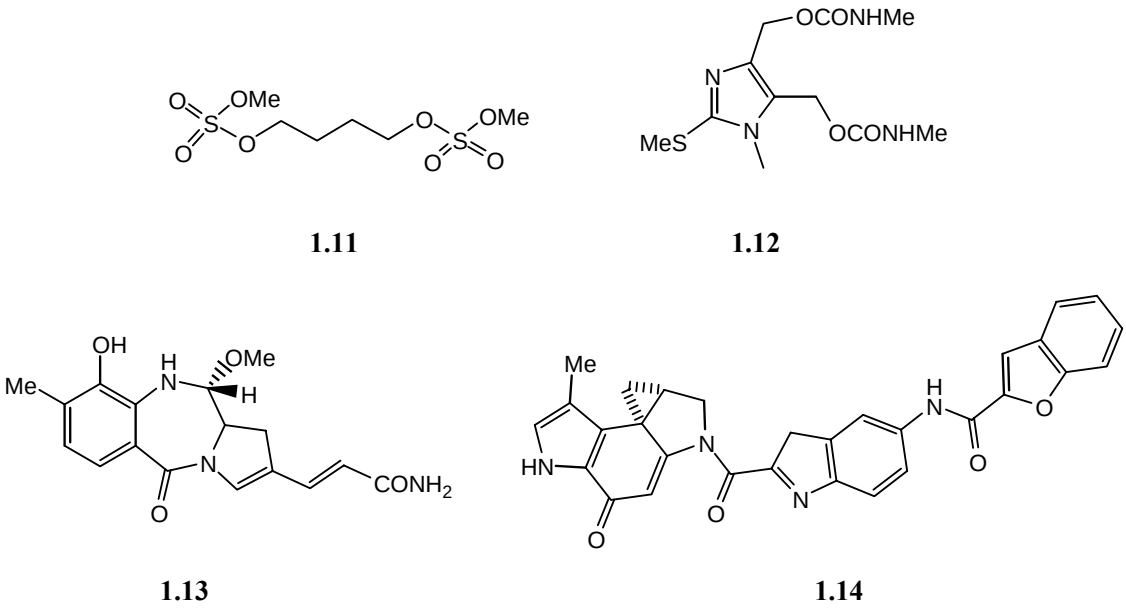
1.2.2.1. Nitrogen Mustards

DNA alkylating agents have played an important part in cancer chemotherapy since the introduction of the nitrogen mustards [*N*,*N*-bis(2-chloroethyl)amines] more than fifty years ago.[37] The first nitrogen mustard used in therapy was mechlorethamine (**1.7**), and the related compounds aminochlorambucil (melphalan) (**1.8**), chlorambucil (**1.9**) and cyclophosphamide (**1.10**) (Figure 1.9) remain in use today. Other DNA alkylating agents of importance are the bismethanesulfonates (*e.g.* **1.11**), the pyrrolizidines (*e.g.* **1.12**), the pyrrolobenzodiazepines (*e.g.* **1.13**) and the cyclopropylpyrroloindoles and their precursors (*e.g.* adozelesin, **1.14**) (Figure 1.10). The first three classes generate DNA crosslinks as the cytotoxic lesion,[38,39] with the latter two being monoalkylators.[40] Mechanism of DNA alkylation of the mustards will be discussed in the next section.



1.7 **1.8** **1.9** **1.10**

Figure 1.9. Some nitrogen mustards



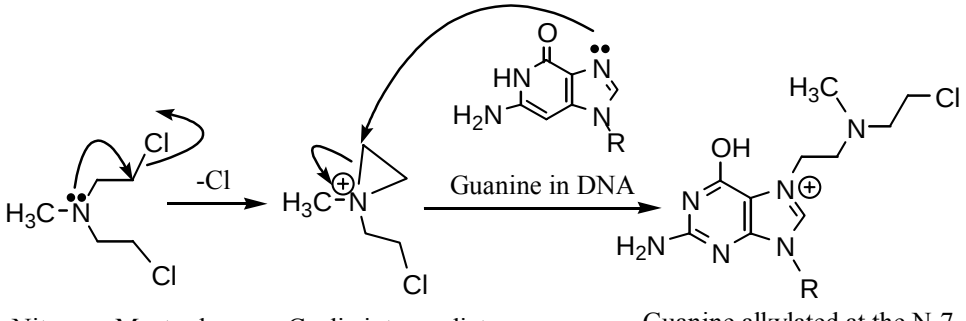
1.11 **1.12**

1.13 **1.14**

Figure 1.10. Some examples of DNA alkylating agents.

1.2.2.1.1. Mechanism and Selectivity of DNA Alkylation of the Mustard Moiety

Mustards alkylate DNA in a two-step sequence involving formation of a cyclic cationic intermediate, followed by nucleophilic attack.[35,37] The mechanism of the action of the N-mustard mechlorethamine, **1.7**, is shown in Scheme 1.2



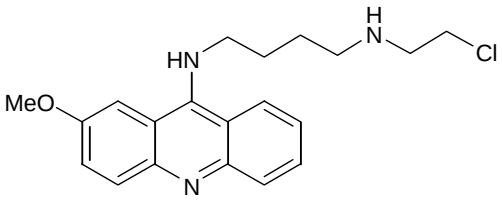
1.7

Scheme 1.2. General mechanism of alkylating DNA by nitrogen mustards.[41,42]

The overall reactivity correlates closely with the basicity of the nitrogen (electron-donating substituents on aromatic mustards increase reactivity).[43,44] For aliphatic mustards the rate-determining step is an S_N2 reaction of the aziridinium cation on DNA.[36] Details of the mechanism are not as clear for the less basic aromatic mustards.[45] The regioselectivity of alkylation of DNA by "simple" nitrogen mustards such as **1.9** is governed largely by the electronic and steric properties of the DNA. This results in a high degree of alkylation in the major groove at the N7 position of guanine,[46] which is the most accessible and has the lowest electrostatic potential, with much lesser amounts of alkylation at the N3 position of adenine in the minor groove.[47-49]

1.2.2.1.2. Examples of DNA-Targeting of Nitrogen Mustards

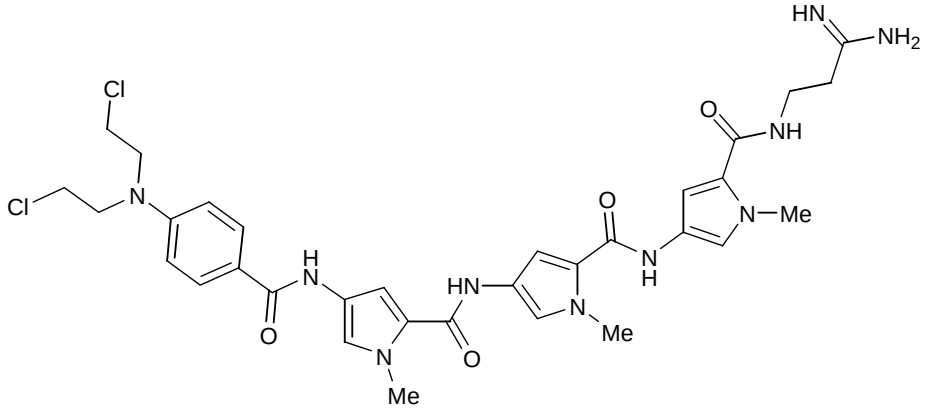
Compound **1.15** (Figure 1.11) is a DNA-intercalating acridine that also alkylates DNA at both guanine and adenine sites.[46,50,51]



1.15

Figure 1.11. A DNA-intercalating acridine

The oligopyrrole antibiotics netropsin and distamycin A have been well-documented by a number of techniques[52,53] as AT-specific reversible minor groove binders. Polypyrrole analogues of distamycin with attached mustards showed highly specific alkylation at adenines in runs of adenines and broad-spectrum solid tumour activity, and the benzoic acid mustard derivative tallimustine, **1.16**, (Figure 1.12) was selected for further development.[54] This compound is a very sequence- and regioselective alkylator, reacting only by monoalkylation at the N3 position of the 3′-adenine in the sequence 5'-TTTTGA-3', with a single base modification in the hexamer completely abolishing alkylation.[55,56]



1.16

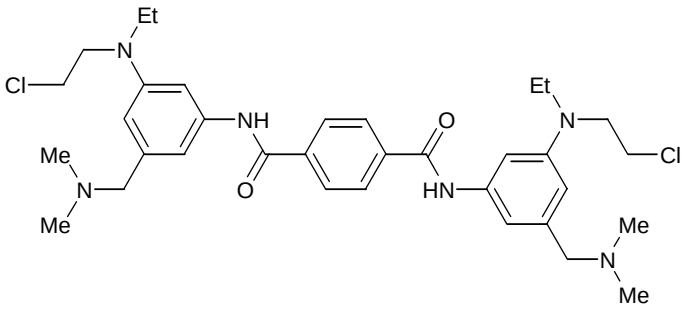
Figure 1.12. The benzoic acid mustard derivative tallimustine.

The polybenzamide motif first used in the anticancer terephthalanilides[57] has been explored as a minor groove binding moiety.[58,59] These compounds are notable for their use of two separate monofunctional mustards, both of which appear necessary for maximum cytotoxicity.[59]

Some studies showed that analogues with two alkylating functions (*e.g.* compound **1.17**) (Figure 1.13) are the most cytotoxic, with **1.17** being 1000-fold more potent than the clinical mustard chlorambucil against P388 leukemia in culture, as

well as being more potent *in vivo*.[59] Monofunctional analogues were also significantly more cytotoxic than chlorambucil, despite bearing much less reactive mustard species.

These results support the concept that targeting nitrogen mustard alkylating agents to DNA by attachment to DNA-affinic carriers can greatly enhance cytotoxicity due to alkylation, and that even for such DNA-targeted mustards, crosslinking is a more toxic event than monoalkylation. Close analogues of **1.17** differing only in their radius of curvature, appear to alkylate and crosslink DNA in similar fashion, yet have widely differing cytotoxicities. The most cytotoxic compound, **1.17,** possesses a geometry most complementary to that of duplex DNA, suggesting that the most toxic lesions are those which result in least DNA distortion, thus being less easily recognised by DNA repair systems.[59]



1.17

Figure 1.13. A monofunctional mustard

1.3. Photodynamic Therapy and Photosensitizers

The drugs which require activation *in situ* by visible or near-IR light to elicit their cytotoxicity are so called photosensitizers.[60] Among their advantages is their ability to localize the toxicity by photoirradiation to a selected site, thus avoiding the complication of systemic toxicity common to many chemotherapeutic agents. Another advantage is their ability to achieve a relatively high therapeutic ratio at most sites

because of factors such as selective tumour uptake-retention and selective photobleaching (*i.e.* inactivation) in normal tissues achievable with certain photosensitizers. The disadvantages include limited depth of effect because of limited light penetration into tissues and the necessity to use fairly expensive, cumbersome laser systems of variable reliability to activate photosensitizer.[60] To some extent, each of these drawbacks has been mitigated (*e.g.* by using interstitial light delivery to deliver light at greater depth, and by recent technical developments in medical laser systems). However, except in certain extracorporeal applications, PDT[61,62] as currently practiced is not capable of treating widespread metastases or systemic cancers. Photodynamic reactions in biological systems have been known for nearly a century, beginning in 1900 when Raab[63] described the killing of paramecium exposed to acridine and light. Subsequent studies[61,62,64-68] demonstrated that this phenomenon occurred with several other light-absorbing materials such as eosin and chlorophyll. Subsequently, oxygen was found to play an essential role. The photodynamic process is considered to be a type II photochemical reaction producing singlet oxygen; that is,

1	$Sen + h\upsilon$	⟶	$^1Sen^*$	*(Excitation)*
2	$^1Sen^*$	⟶	$^3Sen^*$	*(Intersystem Crossing)*
3	$^1Sen^*$	⟶	Sen	*(Internal Conversion)*
4	$^1Sen^*$	⟶	$h\upsilon^1$	*(Fluorescence)*
5	$^3Sen^* + O_2$	⟶	$Sen + {}^1O_2{}^*$	*(Energy Transfer)*
6	$^1O_2{}^* + Biological\ Target$	⟶	*Oxygenated Products*	

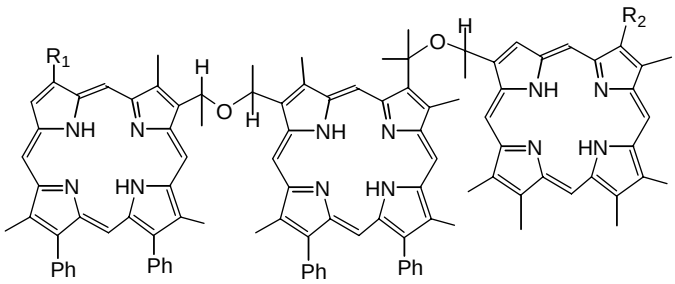
Scheme 1.3

where $^1Sen^*$ is the sensitizer in its excited singlet state, $^3Sen^*$ is the sensitizer in its excited triplet state, and $^1O_2^*$ is the excited singlet molecular state of oxygen. For PDT to be effective *in vivo*, the sensitizer must bind at or near the *biological target*. Thus, in certain cases, electron transfer from the excited sensitizer to oxygen may occur; the process produces superoxide ion (O_2^-) and oxidized substrate[4].

PDT has been developed over the past decade into a useful treatment for several types of solid cancers in man. This unique therapy requires a photosensitizer which is accumulated in tumours and then activated by visible light generally delivered from lasers to the patient through various types of fibers and endoscopes. In Figure 1.14 compound **A** used in photodynamic therapy (PDT) is illustrated.

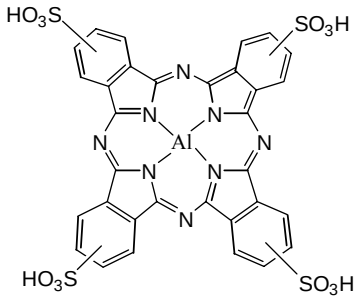
In addition to greater light penetration into tissue, a potential practical advantage of developing photosensitizers for PDT that absorb near 800 nm is the availability of diode lasers in this range.

A series of metalloaluminium naphthalocyanines sulfonated to various degrees on the phenyl groups (**B** in Figure 1.14) was examined by van Lier and co-workers.[69] The number and position of the sulfonated groups were found to be important for *in vitro* reactivity.



$R_1 = R_2 = CH(OH)CH_3$ or $CH{=}CH_2$

A



B

Figure 1.14. A. Hematoporphyrin ether trimer; **B.** Sulfonated chloroaluminium phthalocyanin.[61]

1.4. Hypoxia Selective Cytotoxins: Classification and Mechanism of Activation

The overriding problem in the development of drugs effective against solid tumours is to differentiate between neoplastic cells and normal tissue at a level other than the cytokinetic level (almost all cytotoxic drugs in clinical use are DNA-damaging antiproliferative agents, which are most effective against high growth fraction tumours such as leukemia). Fundamental differences exist between normal and tumour tissues at the level of tumour physiology.

In recent years, a growing interest has occurred in the design and development of bioreductive drugs which can undergo selective metabolic reduction in the absence of oxygen to generate cytotoxic agents (hypoxia-selective-cytotoxins, HSCs).[44,70-80] These compounds have been designed to recognize and exploit the differences between solid tumours and normal tissues, and a number of reviews have been published.[36,81-83]

The imperfect blood-vessel network that develops in solid tumours rapidly leads to the presence of a variable but significant proportion of hypoxic cells.[71-73,84] Figure 1.15 shows the hypoxic region in a cancerous tissue.

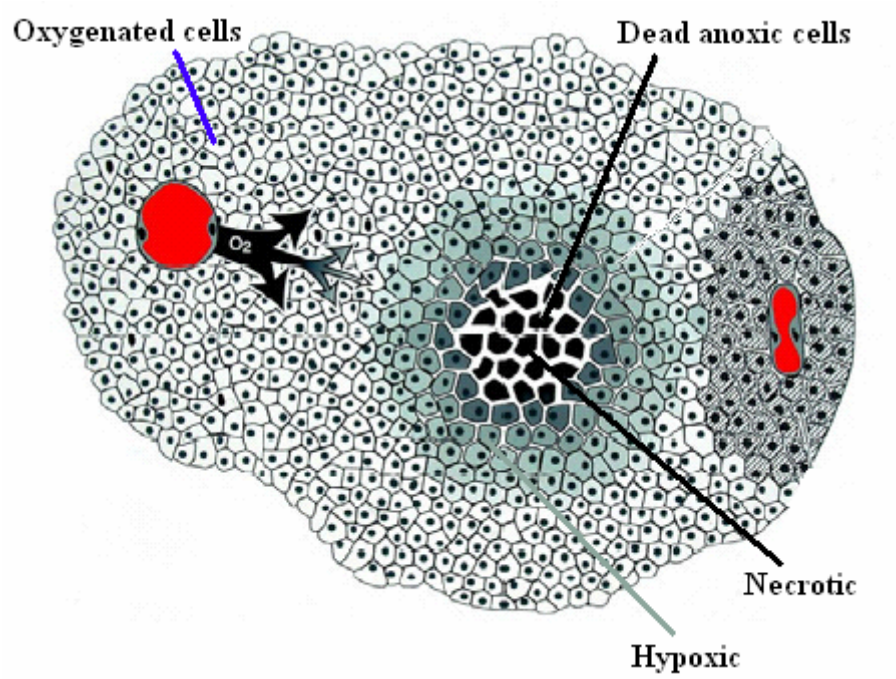


Figure 1.15. The hypoxic region in a cancerous tissue.[85]

HSCs exploit a unique aspect (prolonged hypoxia) of the physicochemical environment of cells inside solid tumours. Thus, to an unprecedented extent, such drugs can be designed a priori by using chemical principles, a situation that holds great appeal for medicinal chemists.

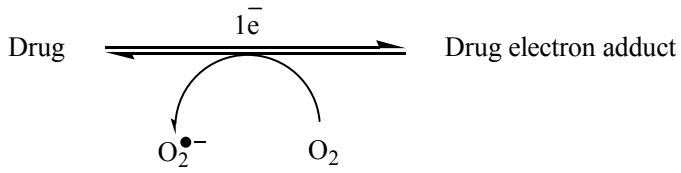
Hypoxic cells are resistant to radiotherapy, mainly because oxygen is required to convert the initial DNA free radicals formed by ionizing radiation into strand breaks (primarily through hydroperoxide species). Such cells are often resistant to drugs, largely because of their slow division rates and their extreme distance from blood vessels.[71,73,86]

The relative resistance of hypoxic cells to chemotherapy has been documented, for a variety of reasons.[87] A few drugs such as the radical generators bleomycin and neocarzinostatin[60] work by generation of DNA radicals, which then generate DNA breaks following reaction with oxygen.[88,89] More generally, hypoxic cells are usually noncycling, because of a lack of nutrients as well as oxygen.[90] This property makes

them resistant to the majority of clinical antiproliferative drugs, which are selective for cycling cells.

Although these factors combine to make the hypoxic cells in solid tumours particularly resistant to conventional anticancer drugs, at the same time the hypoxic microenvironment offers an alternative target.[60]

HSCs need to undergo rapid and selective metabolism in hypoxic environments. Rather than using a mechanism that is exclusive to hypoxic cells, selectivity is normally achieved by utilizing a reductive mechanism that occurs in all cells but that is (at some early point) reversible by molecular oxygen in oxygenated cells. In almost all HSCs studied to date,[44,70-80] the activation process has been a bioreductive one. A wide variety of enzymes exist[83,91] that can add electrons to substrates of suitably high reduction potential (Scheme 1.4).

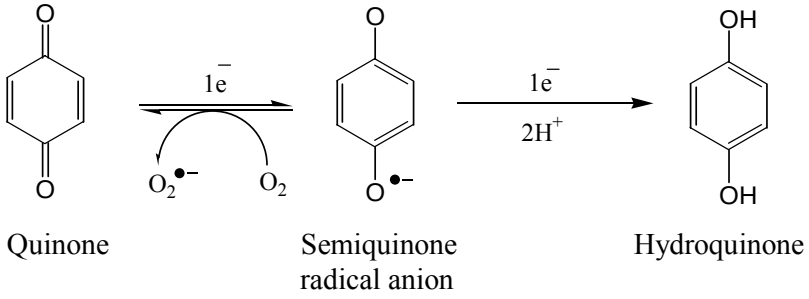


Scheme 1.4. Enzymatic production of a drug electron adduct by adding an electron to the drug.

Another desirable property of the ultimate metabolites is that they have a half-life sufficiently long to permit them to diffuse from the cell of origin. Very reactive species such as nitrenium or carbonium ions, and some radicals, have very short half-lives, reacting primarily with immediately adjacent solvent (water) molecules. Ideally, lifetimes will be in the range of many seconds, which would allow the metabolite to diffuse from the cell in which it is activated to surrounding cells. Such compounds will not just kill the hypoxic cells but make them foci for the production of cytotoxic

species that can affect surrounding tumour cells. The most desirable proximal toxic metabolite of the prodrug is a DNA-alkylating agent, because such agents are the most likely to be effective against non-cycling cells.

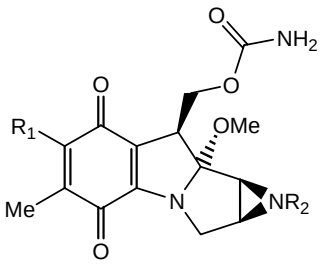
Quinones undergo reduction by cellular enzymes (*e.g.* cytochrome p450 reductase) in two one-electron steps (Scheme 1.5).

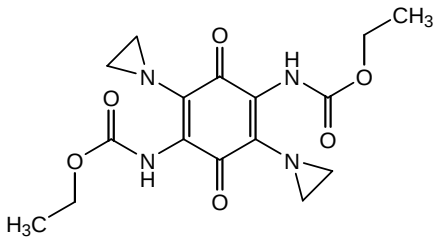


Scheme 1.5. Enzymatic reduction of quinone.

Because the semiquinone radical anion is capable of being scavenged by molecular oxygen in normal tissues, quinones satisfy the theoretical requirement for hypoxia selective reduction.[60]

A number of quinone based drugs have been studied. Mitomycin C, **1.19**, (Figure 1.16) shows variable hypoxic selectivity among different cell lines.[92] Profiromycin, **1.20**, the *N*-methylaziridine analogue, has superior selectivity because of lowered aerobic cytotoxicity[93] (possibly because of lowered aziridine reactivity) *in vitro* and *in vivo*[94] and is now in clinical trial as an HSC. A number of synthetic aziridinylquinones (*e.g.* AZQ, **1.21**, in Figure 1.16), previously evaluated as conventional anticancer drugs, have been studied as potential HSCs, with some showing modest selectivity.[95]



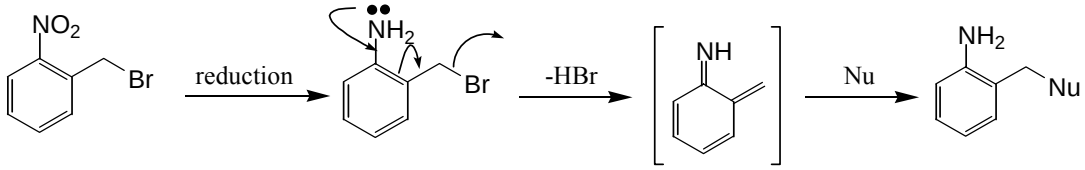


$R_1 = NH_2$; $R_2 = H$: mitomycin C, **1.19**
$R_1 = NH_2$; $R_2 = Me$: porfiromycin, **1.20**

AZQ, **1.21**

Figure 1.16. Quinone based drugs with different selectivity.

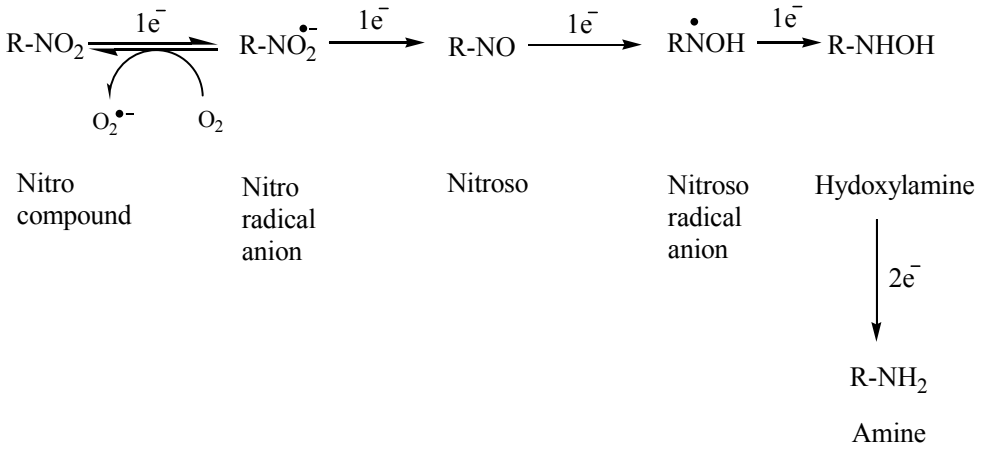
Another class of HSCs, although nominally nitro-aromatics, has been proposed to proceed *via* quinone-type intermediates. Nitrobenzyl compounds bearing efficient leaving groups such as halides and carbamates in the *ortho* position (*e.g.* Scheme 1.6) show modest hypoxic selectivity in cell culture and are postulated to proceed *via* reactive quinoneimine methides.[96]



(nitro compound)

Scheme 1.6. Nucleophilic reaction incorporating a nitro-aromatic compound.

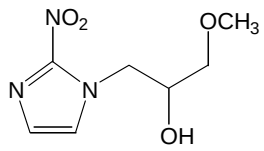
Nitro aromatic compounds are reduced in cells by a number of flavoprotein enzymes[91] that effect stepwise addition of up to six electrons to give the amine, proceeding through the nitro radical anion, nitroso, and hydroxylamine compounds as identifiable intermediates (Scheme 1.7).



Scheme 1.7. Reduction of a nitro aromatic compound in a cell by enzymes. (R= aromatic system)

Because the initially formed radical anion can be scavenged by molecular oxygen, nitro aromatic as a class also fulfill the basic criterion of having a hypoxia-selective metabolism. Most nitro aromatics that have sufficiently high redox potentials to undergo efficient cellular reduction give products that are not markedly cytotoxic. However, some compounds, which appear to undergo rearrangement on reduction to unstable intermediates, also display hypoxia-selective cytotoxicity.

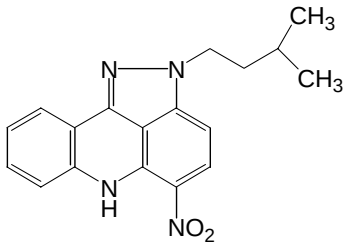
The most studied class is the 2-nitroimidazoles, which were developed as radiosensitizers[60,97] but which also show significant hypoxia-selective cytotoxicity and were the first class of HSCs to be extensively studied (Figure 1.17).[98] Compounds of this type undergo rearrangement following reduction[99,100] to give cytotoxic electrophilic species capable of alkylating DNA.



1.22

Figure 1.17. Misonidazole, **1.22**.

Another class of DNA-binding nitro aromatic HSCs are the 4-nitropyrazoloacridines.[60] These compounds were developed primarily for their solid tumour activity,[101] but some members of the series (Figure 1.18) show significant hypoxic selectivity in cell culture.[102] To what extent the latter feature contributes to their antitumour activity is not known.

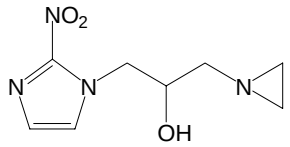


1.23

Figure 1.18.[60]

A large number of classical cytotoxins in clinical use today are classed as alkylating agents, that is compounds that are able to introduce alkyl groups into biomolecules under physiological conditions (pH 7.0-7.4, 37^{o}C, aqueous solution).[36] One subset of the alkylating agents are the N-mustards, of which mechlorethamine is an archetypal example. (Scheme 1.2)

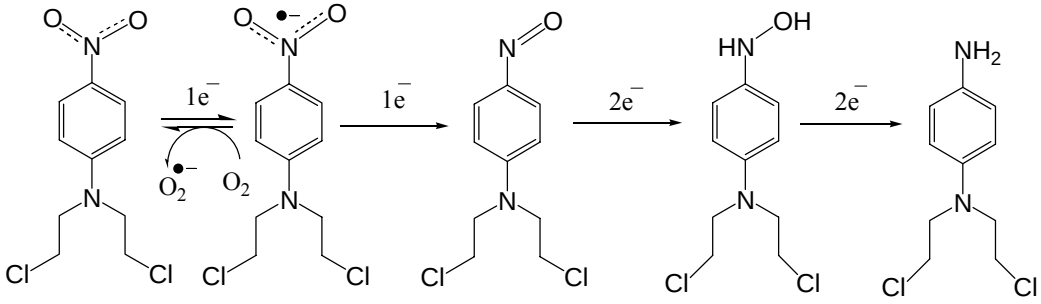
A number of "dual-function"[60] nitro aromatics containing an attached alkylating function have been evaluated, the most studied being the 2-nitroimidazolylaziridine (RSU-1069, **1.24**) (Figure 1.19).[103,104] This compound does have much higher cytotoxicity than non-alkylating 2-nitroimidazoles,[103] but also shows enhanced hypoxia selectivity. This example serves as an illustration of the advantage of providing metabolites of enhanced toxicity following activation.



1.24

Figure 1.19. RSU-1069, **1.24**.

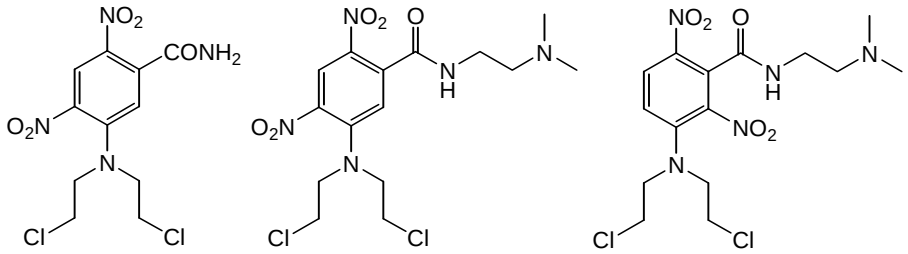
A further class of hypoxia selective cytotoxins containing N-oxides which have received a large amount of attention are nitrophenyl mustards.[42,60] These molecules in general do not rely on the fragmentation of the prodrug to produce the cytotoxic species, although in some cases fragmentation to produce a reactive radical and an alkylating species does occur. For the majority of these species activation of cytotoxin is through an increase in the electron density on the mustard nitrogen, achieved by the biological reduction of the electron-withdrawing nitro-group to form the electron-donating hydroxylamine and amino group as shown in (Scheme 1.9).[105]



Scheme 1.9. Activation of nitrophenyl mustard hypoxia selective prodrugs.

As the cytotoxicity of mustards correlates to the electron density on the nitrogen atom,[42,97] the toxicity of the mustard on the prodrug itself is suppressed but its reduced analogue is cytotoxic. The initial one electron reduction product may be back oxidised to reform the prodrug in oxygen rich cells.

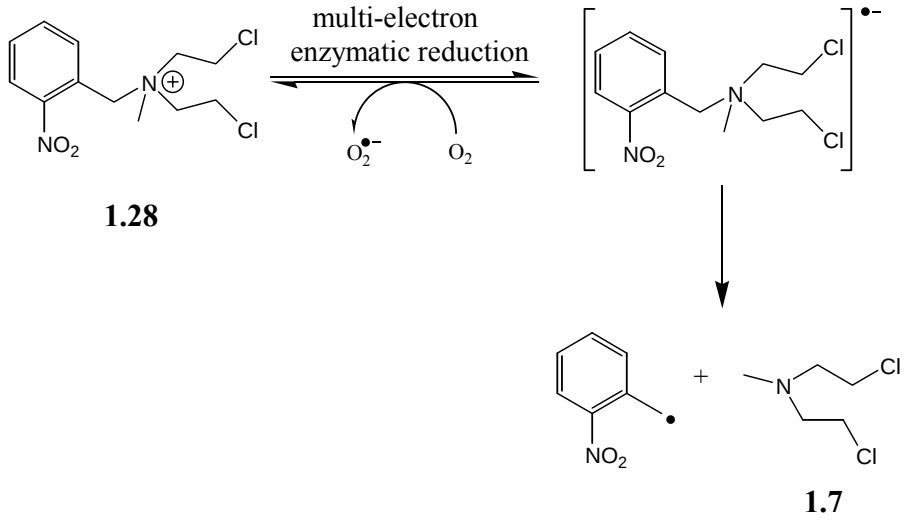
Whilst the hypoxia selectivity of the simple model compound *N,N*-bis(2-chloroethyl)-4-nitroaniline (Scheme 1.9) was limited by its low reduction potential,[97] the synthesis of a number of analogues with a reduction potential within the range for efficient biological reductase reduction and enhanced solubility has been achieved. The parent compound 5-[*N,N*-bis(2-chloroethyl)amino]-2,4-dinitrobenzamide, **1.25**, and its dimethylaminoethyl derivative, **1.26**, as well as 3-[*N,N*-bis(2-chloroethyl)amino]2,6-dinitrobenzamide, **1.27**, (Figure 1.20) displayed activity *in vitro* and *in vivo* but were not selective for hypoxic cells *in vivo*.[106]



1.25 **1.26** **1.27**

Figure 1.20. The compounds 5-[*N,N*-bis(2-chloroethyl)amino]-2,4-dinitrobenzamide, **1.25**, and its (dimethylamino)ethyl derivative, **1.26**, and 3-[*N,N*-bis(2-chloroethyl)amino]-2,6-dinitrobenzamide, **1.27**, synthesized as nitrophenyl mustard prodrugs.

In contrast to the mechanism of action of these nitrophenyl mustards, reduction of the stable[107] nitrophenyl mustard prodrug *N,N*-bis(2-chloroethyl)-*N*-methyl-*N*-(2-nitrophenyl)ammonium chloride, **1.28**, results in its fragmentation to form the readily diffusable cytotoxic species mechlorethamine and a benzyl radical according to Scheme 1.10.[108,109] *In vitro*, this molecule showed a 1000-fold selectivity for hypoxic cells whilst *in vivo* a low but significant activity was observed.[107]

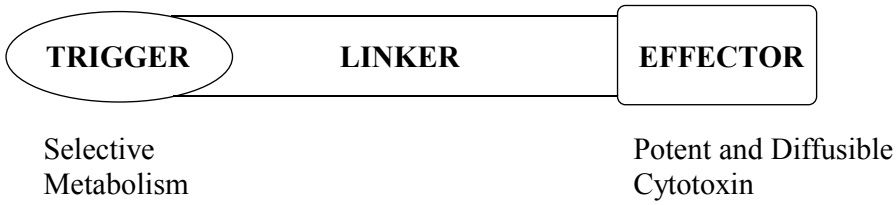


1.28 **1.7**

Scheme 1.10. Mechanism of activation of the hypoxia selective prodrug **1.28**.

1.4.1. General Design Principles for Hypoxia-Activated Prodrugs

Regardless of the chemistry chosen, there are some general design principles that are important for the design of hypoxia-activated prodrugs. In considering these, it is helpful to consider a generic design for prodrugs, in which they are comprised of three (not always distinct) domains; a 'trigger' and 'effector', joined by a 'linker' system[73,110] (Scheme 1.11).



Scheme 1.11

The intact prodrug must be non-toxic and able to diffuse efficiently from the blood vessels through tumour tissue, since the target cells in the case of hypoxia-

activated prodrugs are generally remote from blood vessels. Recent work with a new multi-cellular layer (MCL) model of extravascular diffusion[111] has demonstrated that drug lipophilicity is an important parameter in controlling diffusion rate, together with adequate stability and a lack of macromolecular binding (which lowers the free drug concentration).

The trigger must undergo efficient and selective metabolism in the hypoxic cells, to generate a toxic effector. Hypoxia-activated prodrugs are often classified by the nature of the trigger unit.[73]

The effector should be potent (there may be limits on the capacity of the activating mechanism), and be able to efficiently kill cells in all proliferative states (many hypoxic cells are not cycling).[73] Deoxyribonucleic acid alkylating or breaking agents fulfil this requirement better than antimetabolites or topoisomerase inhibitors. The effector should ideally also have the ability to back diffuse from the hypoxic cell to kill surrounding (maybe oxygenated) tumour cells as well, requiring good diffusion properties and an appropriate lifetime (suggested to be from many seconds to a few minutes). This 'bystander effect' (Figure 1.21) is important, since hypoxic cells are always only a small proportion of the cells in a tumour.[73]

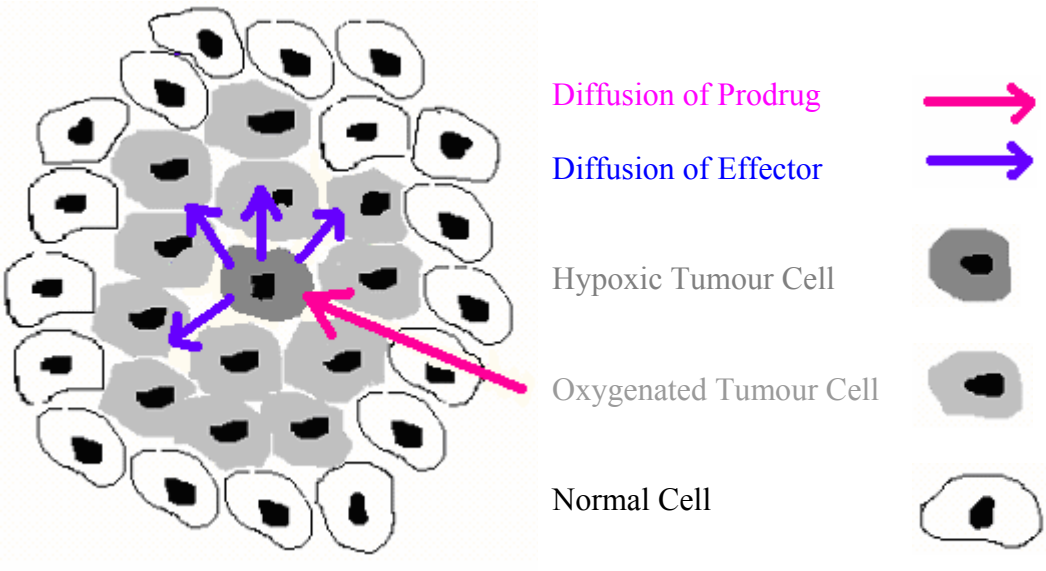


Figure 1.21. Hypoxic cells in a tumour.

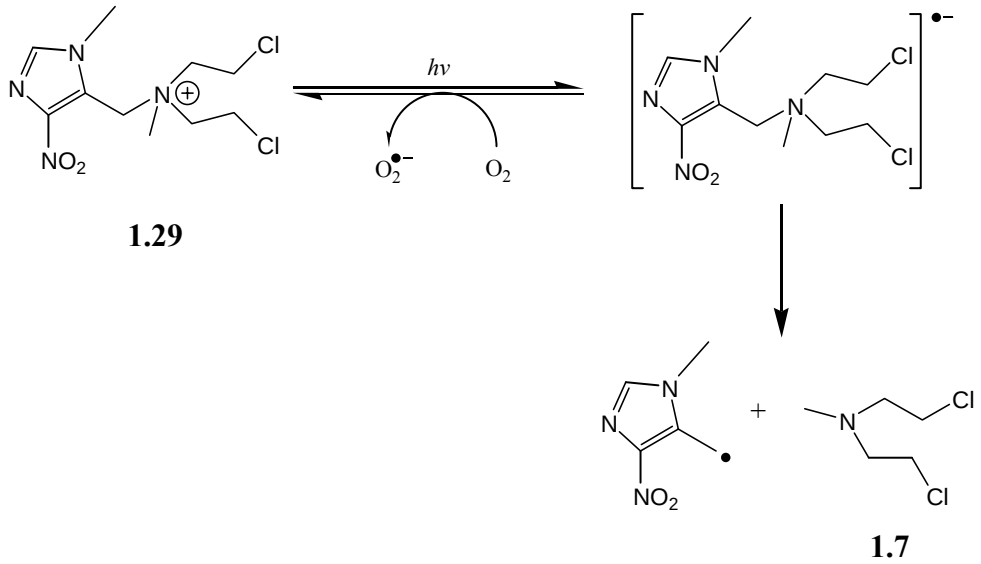
The linker may be a separate, identifiable domain, or a mechanism. It must deactivate the effector in the intact prodrug, yet must rapidly transmit an activating signal on metabolism of the trigger[73,112,113].

1.4.2. Photo-Activated Cytotoxins

The radiolysis of water by ionizing radiation produces reducing species (primarily the aquated electron).[73] These, can in principle, be used instead of reductive enzymes to activate prodrugs in an oxygen-inhibited manner, since both the initial reducing species and the subsequent drug one-electron adduct are capable of being scavenged by molecular oxygen in normal cells.[71,73]

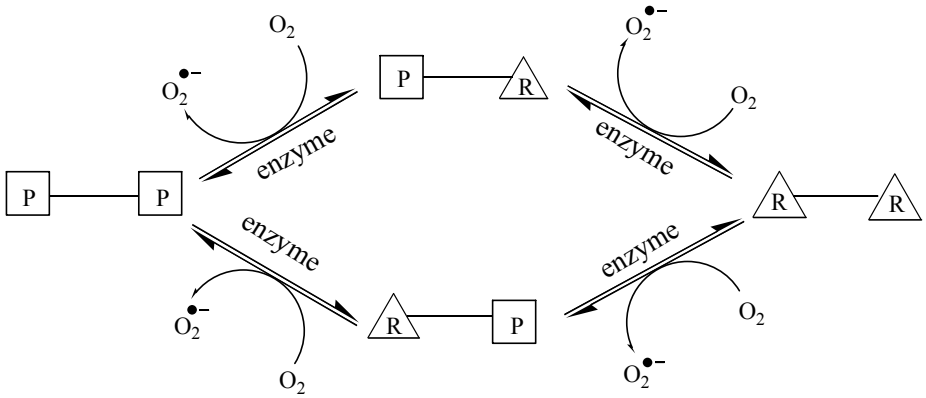
The process in Scheme 1.10 can also take place using irradiation instead of reductive enzymes. Denny and his research group have shown that the quaternary

ammonium salts such as **1.29** are suitable triggers releasing the drug mechlorethamine, **1.7**, under irradiation, *via* a nitro radical (Scheme 1.12).[71,114]



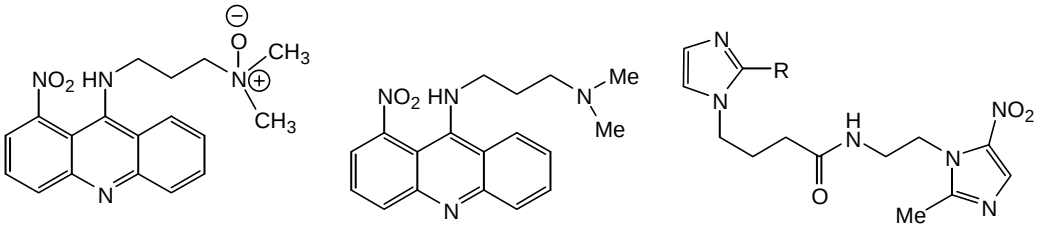
Scheme 1.12

One strategy recently shown[115] to be successful in increasing hypoxia-selective cytotoxicity dramatically beyond this level is the use of two reducible centres in the molecule, both of which must be reduced (by independent, oxygen inhibitable processes) to develop full hypoxic cytotoxicity[60] (Scheme 1.13).



Scheme 1.13. P=Prodrug; R=Reduced form

Thus, nitracrine N-oxide, **1.30**, (Figure 1.20) shows a selectivity of 1000-1500-fold in cell culture, making it by far the most hypoxia-selective compound yet reported.[116] Reduction of the N-oxide generates a cationic side chain, increasing DNA binding substantially and generating the known HSC nitracrine, **1.31**, (Figure 1.20), which then forms very toxic DNA adducts upon additional (independent) reduction of the nitro group.



1.30 **1.31** R = NO_2, **1.32**
R = H, **1.33**

Figure 1.20. Hypoxia cell selective compounds.

Another example of bisbioreductive agent is the bisnitroimidazole **1.32** (Figure 1.20).[75] Although, the closely related mononitro compound **1.33** in Figure 1.20 with one reducible centre has similar selectivity to that of misonidazole (*ca*. 10-fold), the bisnitro compound **1.32** is 100-150-fold more selective. The mechanism of action has not been determined but does not seem to involve DNA adduct formation because, unlike RSU-1069, **1.24**, (Figure 1.19), the compound shows equal hypoxia selectivity in both DNA repair proficient and DNA repair deficient cell lines.[75]

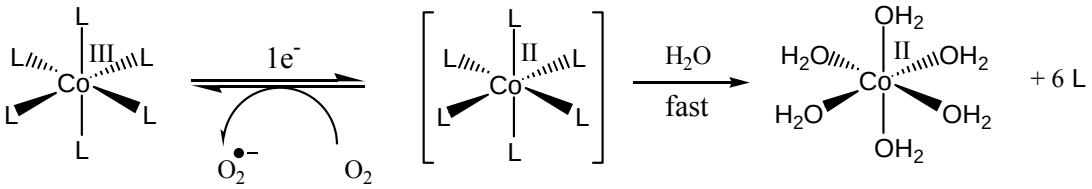
1.4.3. Transition Metal Complexes as Photo-Activated Cytotoxins

A theoretically attractive alternative chemistry for hypoxia-selective deactivated mustards is the use of transition-metal complexes for which only a one-electron reduction is possible.[117] Metal complexes may be treated similarly to organic complexes in their selective accumulation and comparative biochemistry. For example, the requirement of essential metals for cell growth means that alteration of intrinsic metal concentration may preferentially affect the growth of the invading organism.[1]

Various metals form substitutionally inert coordination complexes with nitrogen ligands: Cobalt(III) complexes containing nitrogen mustard ligands are of particular interest. The cytotoxicity of mustards depends on the electron density on the mustard nitrogen, which controls its alkylating reactivity. The nitrogen lone pair is no longer available upon metal coordination. The d^6 low-spin electronic configuration of octahedral Co(III) complexes $[CoL_6]^{3+}$ renders them kinetically inert.

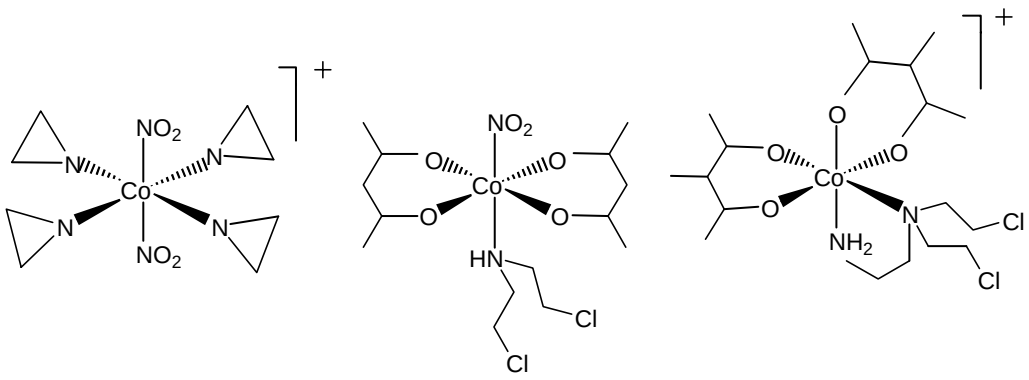
For example, the Co(III) complex of $[Co(NH_3)_6]^{3+}$ has a half-life for ligand displacement of 6×10^9 s[97] and the ammonia ligand would be displaced only very slowly. Since the Co(III)-Co(II) reduction potential can fall in the range of cellular reductants (-200 to -400 m V *vs* NHE), chemical or metabolic one-electron reduction of the inert Co(III) complexes would be expected. The resulting labile Co(II) species $[CoL_6]^{2+}$ would undergo very facile ligand substitution with water, releasing the cytotoxic free nitrogen mustard and $[Co(OH_2)_6]^{2+}$. In other words, one electron reduction to a Co(II) complex results in enormous labilization of such ligands.[106]

Depending on the ligands, hypoxia-selective metabolism is therefore possible, with ligand displacement by water to form the very stable hexaaqua Co(II) cation competing (in oxygenated cells) with reoxidation of the Co(II) complexes by molecular oxygen (Scheme 1.14).[60]



Scheme 1.14. Reduction of a Co(III) complex.

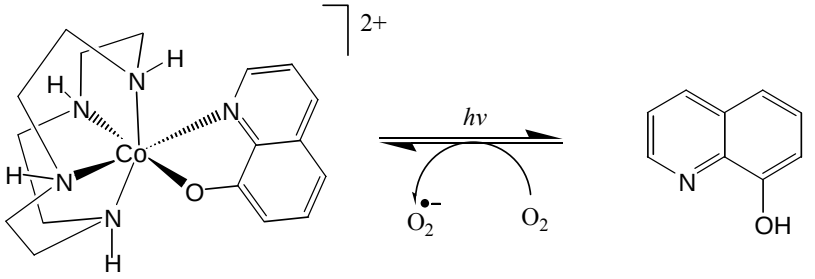
If the ligands (L) are nitrogen-based alkylating moieties, they are likely to be much more cytotoxic as free ligands than when coordinated to the metal, because the electron density on the nitrogen is greatly lowered upon metal coordination. Thus, some studies[107] show that Co(III) complexes of aziridine are greatly stabilized with respect to hydrolysis, even by strong acid. A critical point is whether the reduced $[CoL_6]^{2+}$ complexes can be made sufficiently stable to allow reoxidation in aerobic cells to compete with ligand loss. Studies[107,108] suggested that monodentate alkylating nitrogen ligands could not provide sufficient stability; thus, the aziridine and bis(2-chloroethyl)amine complexes **1.34** and **1.35**, respectively are not hypoxia selective (Figure 1.23). However, a series of bidentate Co(III) mustard complexes has been reported,[109,118,119] where the metal redox potential has been systematically varied, and one of these complexes , **1.36**, (Figure 1.23) shows significant hypoxia selectivity in cell culture. This finding suggests that metal complexes of nitrogen mustards constitute a further class of HSCs.



1.34 **1.35** **1.36**

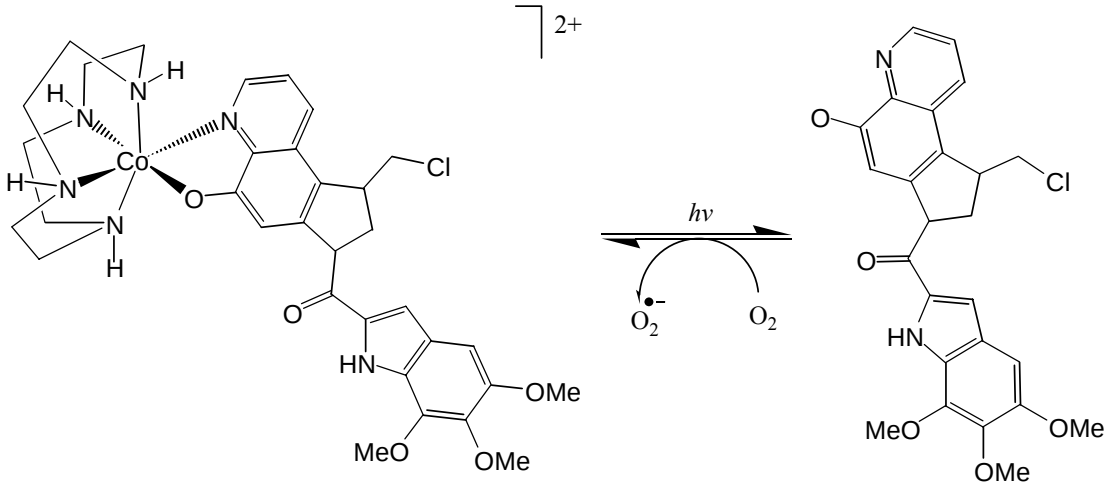
Figure 1.23. Cobalt(III) complexes investigated as hypoxia selective cytotoxins containing an aziridine moiety coordinated as monodentate ligand, **1.34**, a mustard moiety coordinated as a monodentate ligand, **1.35**, or a mustard coordinated as a bidentate ligand, **1.36**.

In seeking more stable polydentate complexes, Denny and his research group found that those containing polyazamacrocyclic auxiliary ligands were also potentially interesting triggers for radiative prodrugs.[120] Of particular interest was the $[(cyclen)Co^{III}(8\text{-HQ})]^{2+}$ (8-HQ = 8-hydroxyquinoline) complex, **1.37** (Scheme 1.15).[71,73,120] This releases the weakly cytotoxic 8-HQ, **1.38,** when irradiated with visible light in formate buffer under hypoxic conditions. While **1.38** is not potent enough as a cytotoxin, complex **1.37** serves as a useful model for complex **1.39** of the much more potent hydroxyazaCBI class of 8-HQ analogues, **1.40**, that Denny and his co-workers developed (Scheme 1.16).[71,73] In their studies, irradiation of **1.39** was able to generate toxicity to cell cultures, thus providing experimental proof of principle for photo-activated cytotoxins.[71,73]



1.37 **1.38**

Scheme 1.15. A Co(III) complex as a photo-activated cytotoxin.



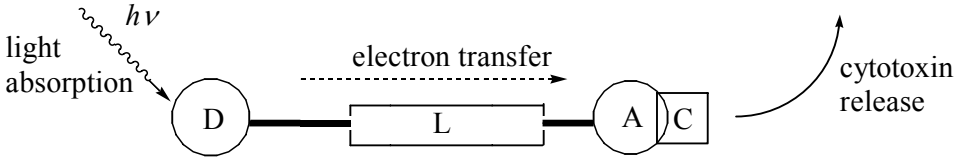
1.39 **1.40**

Scheme 1.16. A Co(III) complex as a photo-activated cytotoxin.

1.5. Research Outline

An alternative approach to the design of more selective drug delivery systems is to use radiation in the UV to visible range to effect the activation of a prodrug. The advantage of this approach is that activation, and hence drug delivery, can be limited to specific localised tumour volumes defined by the irradiation area. This approach may increase the effectiveness of the drug whilst decreasing many of the side effects experienced with current treatments, provided the prodrug is non-toxic to normal cells.[1,42]

This project is centred around the synthesis and study of molecules which are made up of four components, shown schematically in the Scheme 1.17. They will contain an antenna or electron donor unit (D) which will release an electron when it absorbs a photon of the appropriate wavelength. The donor unit (D) becomes electronically excited upon photoirradiation. Photo-induced electron or energy transfer occurs through the linker to the acceptor unit (A).[121,122] The addition of an electron to the acceptor unit will alter its chemistry so that it will release the cytotoxin (C), which is inactive until it is released.



Scheme 1.17. Photoactivated cytotoxin: Photoinduced electron transfer from donor (D) to acceptor (A) through linker (L) causes cytotoxin (C) release.

Possible electron donor units (D) include zinc porphyrin complexes, ruthenium (II)-polypyridine complexes and phenazines, with excitation wavelengths ranging from the red, through blue, to the UV. Clearly, the UV irradiation required for the phenazines is not desirable for biological applications, but it may be the best donor unit for demonstration of the concept as there will be a high driving force for photoinduced electron transfer and the compounds are likely to be less photosensitive. In terms of synthetic accessibility, the ruthenium complexes may be preferred.

Polypyridyl ruthenium complexes are widely used because of the intense absorption in the visible range of the spectra, excellent photochemical stability, and strong emissive character.[123-126] Another important factor which makes Ru a viable choice is the ability to arrange ligands into its octahedral geometry.[123,124,127-135,68-76] Ru(II) is a d^6 metal ion and has a ground electronic configuration of $(t_{2g})^6$, and a one-

electron oxidised form, Ru(III), has the configuration $(t_2g)^5$. In these oxidation states, ruthenium is inert to ligand substitution. In fact, ligands which bridge the metal centre to the acceptor site play an important role. This is because, first, the bridging ligand dictates the whole structure and directionality of the donor-acceptor system; second, with its coordinating sites it contributes to the spectroscopic and redox properties of metal-based units; and third, it controls the electronic communication between the electron donor (D) and acceptor (A). Therefore, the selection of suitable bridging ligands is crucial to obtain a well-designed photoactive drug.[123]

A significant body of research currently exploits the synthesis of photoactive ruthenium compounds, for the study of their photochemical, photophysical, electrochemical properties. These investigations have attempted to design and construct new ligands and their corresponding ruthenium complexes capable of performing useful light-induced functions.[123,129-135] In these ruthenium complexes, a metal-to-ligand charge transfer (MLCT) state of the Ru(II) moiety is responsible for most of its photochemistry.[122,123,136]

In recent years, ligands derived from a modification of 2,2'-bipyridine (bpy), **1.1**, and 1,10-phenanthroline (phen), **1.2**, have been employed for some applications.[122,125-131,133-135] Among a great number of bridging ligands containing groups, these rigid π-conjugated systems are ideal selections because they reduce the bending along and/or rotation around the σ-skeleton of the molecule. In addition, π-electron conjugation over the aromatic parts allows a long-distance, yet sufficiently strong electronic interaction between those units.[123,128]

The acceptor-cytotoxin unit will be based around substitutionally inert Co(III) or Cr(III) complexes containing mustard ligands. Reduction of these complexes will allow release of the mustard since the related Co(II) and Cr(II) complexes will be

substitutionally labile. This kind of chemistry has already been exploited as a potential basis for hypoxic cell selective anti-cancer agents, where the activation is achieved by enzyme mediated reduction within the hypoxic cell.[117,137,138]

It is anticipated that macrocyclic ligands will be used for this part of the molecule, as they form stable and robust complexes and can be readily attached to other molecules.

The properties of cobalt(III) make its complexes an ideal choice of acceptor unit for the photo-activated donor-acceptor concept.

A wide range of linker units are possible and their length and constitution will be used to tune the electron transfer properties of the molecules. In particular, the rate of the back electron transfer of the charge separated state has to be sufficiently slow to allow release of the cytotoxin from the reduced acceptor unit. The rates of electron transfer have been found to depend upon a number of factors, including the distance between the donor and acceptor.[42]

1.6. Donor-Acceptor Systems

One of the most fundamental and biologically important chemical processes is the transfer of charge or energy through matter. Over the past several decades this topic has been the focus of intensive research efforts across a range of scientific disciplines with the result that our understanding of these processes has increased markedly.[121,122,126,139-143] The contribution from the discipline of chemistry has been particularly valuable and has allowed the elucidation of events occurring at the molecular level.

Nature has put electron transfer and energy transfer to use in numerous biochemical pathways to affect various chemical changes; for example, in the photosynthetic reaction in which the capture and storage, in the form of chemical bonds, of radiative energy produced by the sun takes place. Energy capture is achieved by the absorption of radiation by a magnesium-porphyrin chromophore. The absorption leaves the chromophore in an electronically excited state and electron transfer from the chromophore to an electron acceptor creates a charge separation between the chromophore and acceptor. The chromophore, in its positively charged state, is able to accept an electron produced by splitting of water to complete the cycle and leave the photosynthetic centre ready to absorb more radiative energy. Once captured the energy is transferred and used throughout the organism as required.

The effort to understand the photosynthetic pathway has stimulated the development of molecular systems containing discrete donor and acceptor units. The development of the capacity to capture energy from radiation and then affect chemical change has an enormous potential for applications in industry and medicine.

The successful design of molecular devices that exploit the donor-acceptor approach hinges on both the efficient transfer of energy or electrons from the donor to the acceptor as well as the formation of a suitably long lifetime of the charge separated state. Chemical research into the properties of donor-acceptor systems has resulted in the synthesis of many synthetic donor-acceptor systems.[121,122,144-146] During the investigation of these systems it has been established that the arrangement of the donor and acceptor moieties with respect to one another, the solvent, and the properties of the linker have a marked effect on the rate of electron transfer between them and the lifetime of the charge separated state.[49-55,32]

1.6.1. Linkers

Linkers play a 2-fold role: (i) control of the supramolecular structure (in particular, of the intercomponent distances and angles); and (ii) control of the electronic communication between components in the case of through-bond energy or electron transfer.[122] Obviously, rigid spacers, such as those based on aromatic rings [147] bicyclo aliphatic species[148-150] and bridges containing ethynyl groups,[151-158] should be preferred to flexible spacers (*e.g.* aliphatic chains) for structural reasons.[122] From the electronic viewpoints, aromatic spacers allow better communication than aliphatic spacers, but a complete picture of the electronic effects of spacers has not yet been obtained.

In particular, the length of the linker is an important factor for electron transfer between the donor and acceptor occurring through the linker itself (the "through bond" electron transfer mechanism).[159] Investigations of the effect of the linker length on the rate of electron transfer (both to form a charge separated state and the charge recombination process) and the resulting lifetime of the charge separated state have been carried out on a number of systems.[160-163] Detailed investigations on a bichromophoric system containing dimethoxynaphthalene (DMN) as the electron donor and dicyanovinyl as the acceptor unit, shown in Figure 1.24, have shown that an increase in the length of the linker, (in these investigations a norbornylogous bridge) results in an increase of the lifetime of the charge separated state.[161,159,164] The rigid norbornylogous bridge used as the linker allows precise control of the relative orientation and separation of the donor and acceptor.[42]

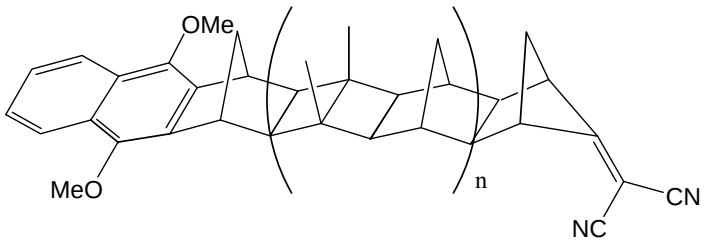


Figure 1.24. Bichromophoric donor-acceptor system used to investigate the effects of linker length.

Through space electron transfer has also been observed in large multichromophoric systems.[42] Ghiggino and Paddon-Row[165] have extended their electron transfer studies of multichromophoric systems to the tetrad **(a)** (in Figure 1.25), containing zinc porphyrin (Pzn) and methyl viologen (MV^{2+}) as the terminal chromophores and dimethoxynaphthalene (DMN) and naphthaquinone (NQ) as intermediate chromophores.[165] This system offers the promise of exploring the relative importance of three competing mechanisms for effecting photo-induced electron transfer from locally excited Pzn donor to MV^{2+} acceptor, namely: (1) superexchange through the bridge, (2) electron hopping through the bridge in which electron transfer takes place, and (3) direct electron transfer between Pzn and MV^{2+}, either through solvent or through space. Intramolecular photoinduced electron transfer through space has been observed in polar solvents for the U-shaped, *syn-syn* isomer **(b)**. In contrast, no electron transfer is observed in a sample containing isomers *anti-syn* **(c)** and *anti-anti* **(d)**. Hence, different isomers of the one system may display remarkably different electron transfer properties, due to the distance constraints of the through space mechanism. One isomer may display quite efficient electron transfer whilst the other does not undergo transfer.[165-167]

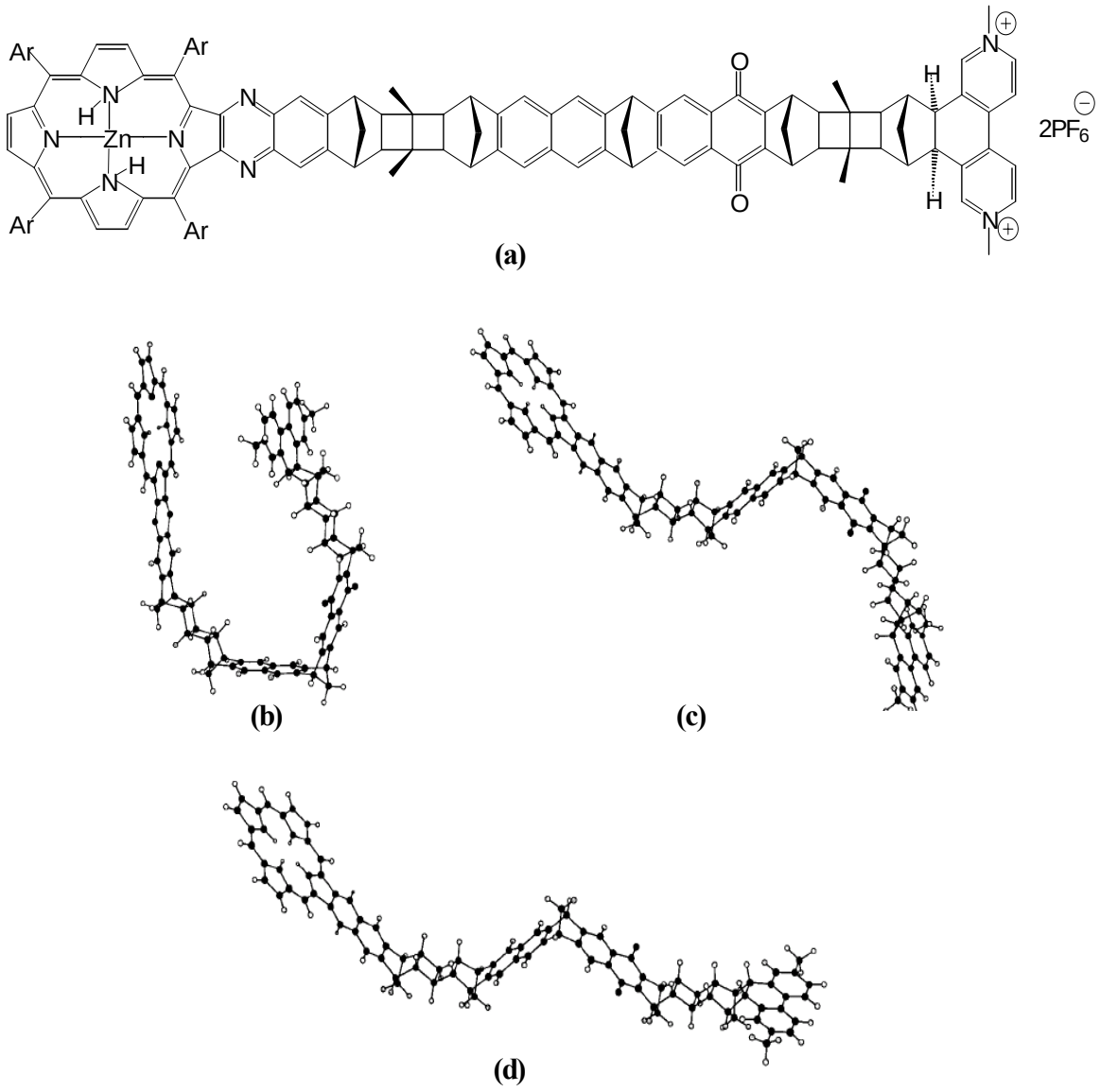


Figure 1.25. Structure of a multichromophoric donor-acceptor system **(a)** that undergoes photoinduced intramolecular electron transfer when the *syn-syn* isomer **(b)** is excited. Other isomers: **(c)** *syn-anti* isomer, **(d)** *anti-anti* isomer. (Ar = 3,5-di-*tert*-butylphenyl).[165]

The use of a metal centre as the electron acceptor unit in donor-acceptor systems has resulted in an increase in the potential number of uses for donor-acceptor systems. The stability of the metal in its different oxidation states and its differing properties in those respective oxidation states give great flexibility and diversity of

potential applications for such systems.[42] Appropriate design and choice of metal used in the donor-acceptor system may result in a system able to carry out a specific task.

The development of donor-acceptor compounds as photoactivated drugs would be of particular significance because in many circumstances there is a pressing requirement for localised drug delivery or activation. An important instance is provided in cancer chemotherapy.

1.7. Synthetic Strategies for Photo-activated Cytotoxin Anticancer Agents

Synthesis and investigation of donor-acceptor systems in which the potential exists for photo-activated ligand release will be a major component of this research work. My objectives are to synthesise models for donor-acceptor systems in order to provide a demonstration of the viability of using donor-acceptor systems as possible photo-activated cytotoxins.

In this thesis, I focus on the preparation and use of ditopic ligands (linkers, L) where the two metal ion binding sites are differentiated by the number of donor atoms in each site, the configuration of the binding site, or the types of donor atoms that are present in the sites. This binding site differentiation offers the prospect of using the different coordination properties of the binding sites to control the regiochemistry of complexation, ensuring that the correct metal ion is incorporated at the desired binding site in the ligand.

This kind of approach has been used before, and a range of heterodinuclear complexes have been prepared using ligands with differentiated binding sites. For example, Fraser *et al.*[168] have prepared ligands containing both an octahedral site and a

square planar binding site that allowed heterodinuclear molecules to be synthesised, Ōkawa *et al.*[169] put together systems containing four and five coordinate sites, and Abe *et al.*[170] have also produced systems with different numbers of donors in each site. Binding sites have also been differentiated by the kind of donor group,[171-180] or by using functional groups such as oximes that are able to bind more than one metal ion.[181,182]

We were particularly interested in minimising the number of stereoisomers that might be produced on synthesis of the heterodinuclear system, and further, we wished ultimately to incorporate a ruthenium(II) ion in one of the sites (donor site, D). As it will be discussed later in this chapter, the terpyridine type ligand fragment appealed because many such ruthenium(II) complexes have been prepared,[122,143-146,156,183-203] the photochemistry of such systems is relatively well understood,[122,126,142,204-206] and the ligand structure ensures a meridional arrangement of the donor atoms, which reduces the number of possible isomers. While the excited state lifetimes for such ruthenium(II) terpyridyl complexes are usually rather short,[207,208] a number of methods have been employed to lengthen the excited state lifetimes of such systems, and these may be incorporated into our eventual system, if required.[144,146,196,198,201,209,210]

As it will also be discussed later, a number of possibilities were considered for the second metal ion binding site (acceptor site, A) and, again, we were interested in reducing the number of stereoisomers that might be produced.

1.7.1. Donor Site (Terpyridine Systems)

Terpyridines were first discovered by Morgan and Burstall.[211] By heating pyridine with anhydrous iron(II) chloride at 340 °C in an autoclave (50 atm) for 36 hours, 2,2':6',2"-terpyridine, **1.3**, was produced (besides bipyridines and other products). It was subsequently discovered that the addition of iron(II) ions to a solution of terpyridine compounds gave rise to a purple colour, giving the first indication for the formation of a metal complex. In the following sections, an overview of the synthesis of various functionalised terpyridine ligands (especially 4'-functionalised terpyridines) is given, followed by a description of the latest achievements in the field of terpyridine complexes, ranging from mononuclear complexes to extended supramolecular architechtures.

A variety of terpyridines, functionalised at different positions, have been prepared over the past few years. Several reviews have been published on the subject. In this thesis, we are particularly interested in terpyridines functionalised at the 4'-position.

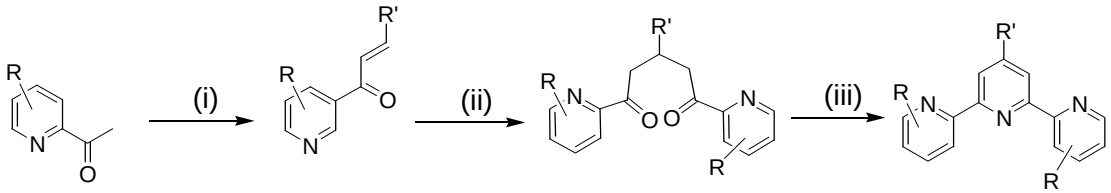
1.7.1.1. Methods for Synthesis of Terpyridine Ligands

Excellent reviews are available on the synthesis of terpyridines and their functionalisation.[203,212-215] In this thesis, we are particularly interested in the functionalisation of 4'-substituted terpyridyl ligands.

Different methods for synthesis of terpyridine ligands are described in the literature. The most used methods in this project are the first two described as follows:

1.7.1.1.1. Kröhnke Methodology

Kröhnke developed condensation methodology leading to oligopyridines.[214] The basis of this reaction is the aldol condensation of 2-acetylpyridine, **1.41**, (or a substituted derivative) with an aldehyde in basic aqueous or alcoholic media to give an α,β-unsaturated ketone or enone **1.42**. Michael addition of the suitable enolate then affords 1,5-diketone **1.43**. Ring closure with ammonium acetate results in the formation of dihydropyridine, which undergoes oxidation to the desired tpy **1.44**. A major advantage of this route is that symmetrical and unsymmetrical terpyridines can be prepared in moderate to good yields. The main disadvantage is, however, that the substituents R' must be aromatic (Scheme 1.18).



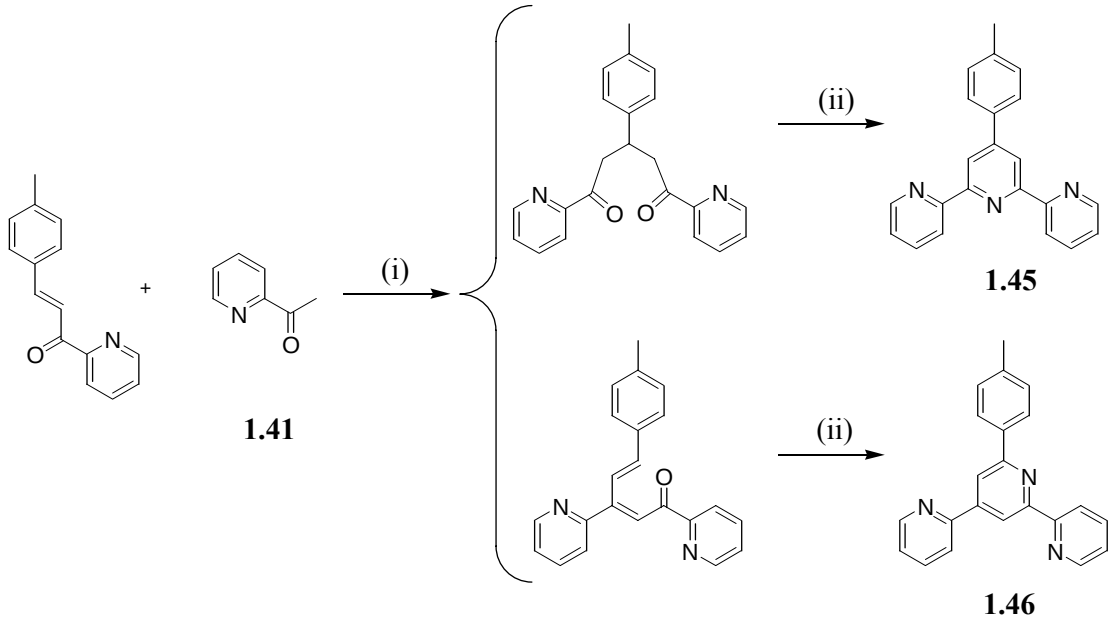
1.41 or its derivative **1.42** **1.43** **1.44**

Scheme 1.18. (i) R'-CHO, NaOH, EtOH, 0 ºC, 60-95%; (ii) N-(1-(2'-pyridyl)-1-oxo-2-ethyl)pyridinium iodide; (iii) $[NH_4^+][OAc^-]$, EtOH; overall yield: 30-80%.

1.7.1.1.2. Collin–Balzani–Sauvage-Constable Methodology for Synthesis of 4'-p-Tolyl-terpyridine (ttp)

In order to obtain large amounts of 4'-*p*-tolyl-terpyridine (ttp),**1.45**, the one-pot Hantzsch synthesis, developed by Case[216] and later by Calzaferri[217] was selected by

Collin-Balzani-Sauvage.[218] Contrary to previously published work,[217] the purification of ttp by crystallization in ethanol or chromatography (alumina or silica gel) proved to be ineffective. In fact, two terpyridine isomers were isolated and characterised. These two isomers can be obtained following a 1,2 or 1,4 Michael addition on the unsaturated ketone intermediate produced by the condensation of the aromatic aldehyde and 2-acetylpyridine, **1.41**. (Scheme 1.19)[218]



Scheme 1.19. One-pot reaction methodology for synthesis of ttp ligand, (i) and (ii) aqueous NaOH, acetamide, ammonium acetate, 120ºC.

The separation of the tridentate ttp ligand and the sterically hindered bidentate ligand 6'-*p*-tolyl-2,2':4',2"-terpyridine, **1.46**, was readily performed by formation of the highly stable $[Fe(ttp)_2]^{2+}$ complex as previously described.[219] The iron(II) complex, treated with H_2O_2 in alkaline solution, yields the free ttp ligand in good yield.

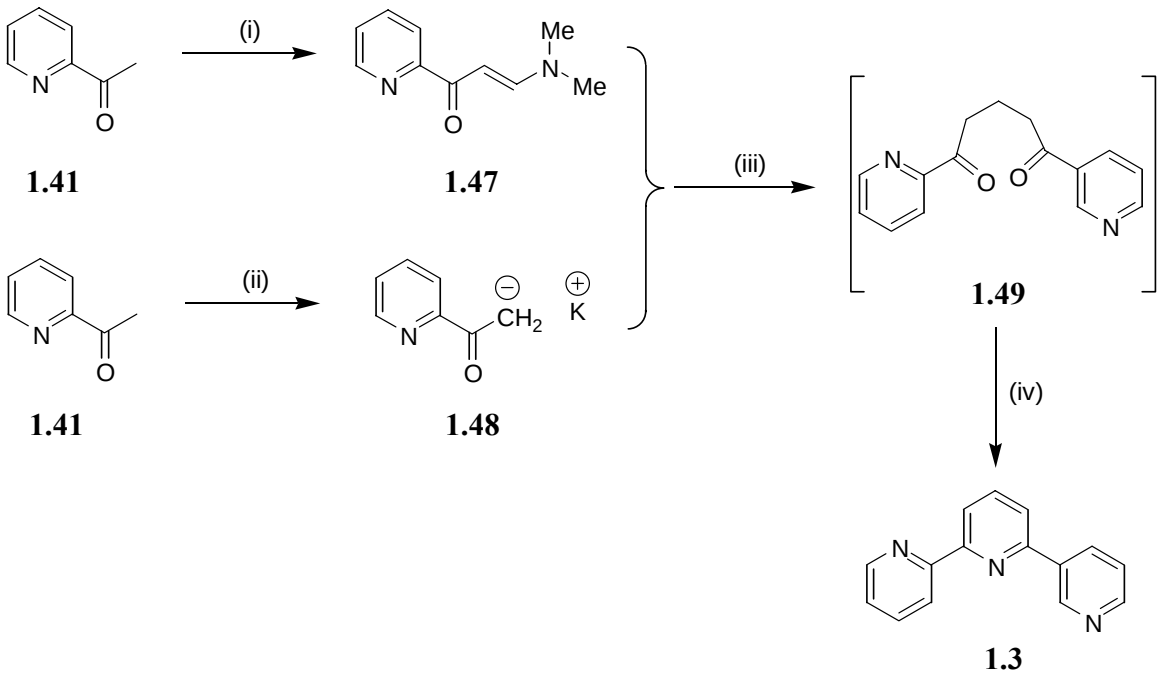
Moore *et al*. recently reported the modification of this method to obtain ttp ligand.[220] They frequently found that during attempts, following Colin-Balzani-Sauvage-Constable method[218,219] to prepare ttp, to extract the minor by-product **1.46**,

the purple acetonitrile solutions of $[Fe(ttp)_2]^{2+}$ were miscible with toluene, and separation of the by-product proved unsuccessful. They were able to successfully isolate the by-product by repeatedly washing the purple $[Fe(ttp)_2]^{2+}$ complex with toluene, and then combining the washings.

Other methods including pyrolysis of hydrazonium salt, Tohda methodology, metal-mediated methodology (nickel-mediated reactions, palladium-mediated reactions (Suzuki reaction or Stille coupling reaction)), and Sauer methodology are also well explained by Fallahpour.[212] It was shown that the Kröhnke methodology is the most convenient way to synthesise aromatic substituted tpy ligands.[212] Using this method, many compounds possessing aromatic substituents at the 4'-position were prepared.[183,187,192,212,213,221-226]

1.7.1.1.3. Jameson and Guise Methodology

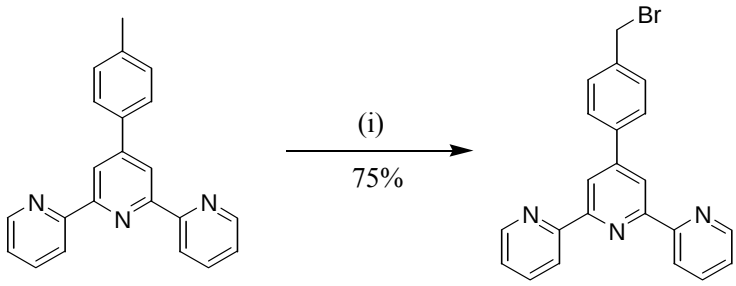
In the Jameson and Guise approach, 2-acetylpyridine, **1.41**, was reacted with *N*,*N*-dimethylformamide dimethyl acetal to give the enaminone **1.47** in high yield.[212] This enaminone **1.47** is condensed with the potassium salt **1.48** to form a 1,5-dione, which is not isolated prior to ring closure by ammonium acetate (Scheme 1.20).



Scheme 1.20. (i) $H(MeO)_2CNMe_2$; (ii) t-BuOK, THF; (iii) THF; (iv) $[NH_4^+][OAc^-]$, HOAc; overall yield: 80%.

1.7.1.2. Functionalisation of 4′-*p*-Tolyl-terpyridine Ligand

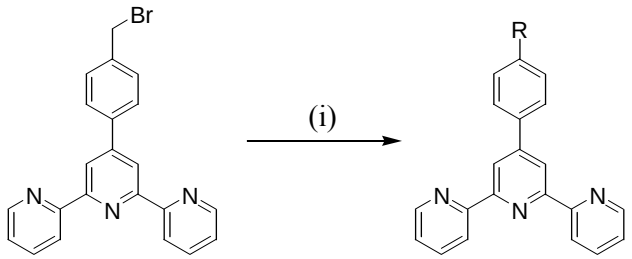
Although several phenyl-substituted terpyridines were synthesised and reported by Araki *et al*.,[226] one of the most versatile ways to functionalise the ttp ligand found in the literature was the radical bromination of its methyl group[193,197,218,220,227,228] (Scheme 1.21).

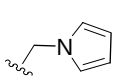
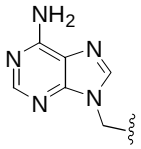
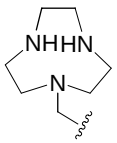
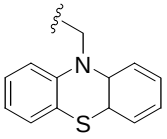
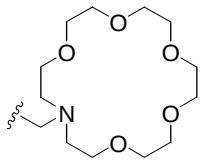
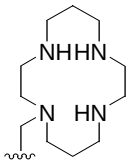
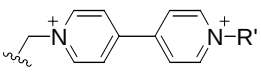
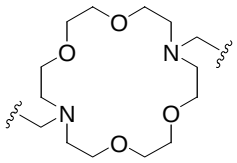
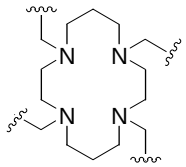
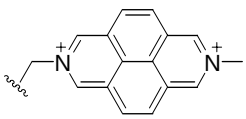


1.45 **1.50**

Scheme 1.21. Radical bromination of ttp ligand. (i) *N*-bromosuccinimide (NBS), dibenzoyl peroxide, dry benzene, *hυ*, reflux, 4hr.

This compound is highly reactive and was reacted under basic conditions with a variety of nitrogen heterocyclic/acyclic/or macrocyclic compounds. (Scheme 1.22)



R	Compound	R	Compound	R	Compound
	**1.51**		**1.55**		**1.58**
	**1.52**		**1.56**		**1.59**
 R' = H, Me, Et	**1.53**		**1.57**		**1.60**
	**1.54**				

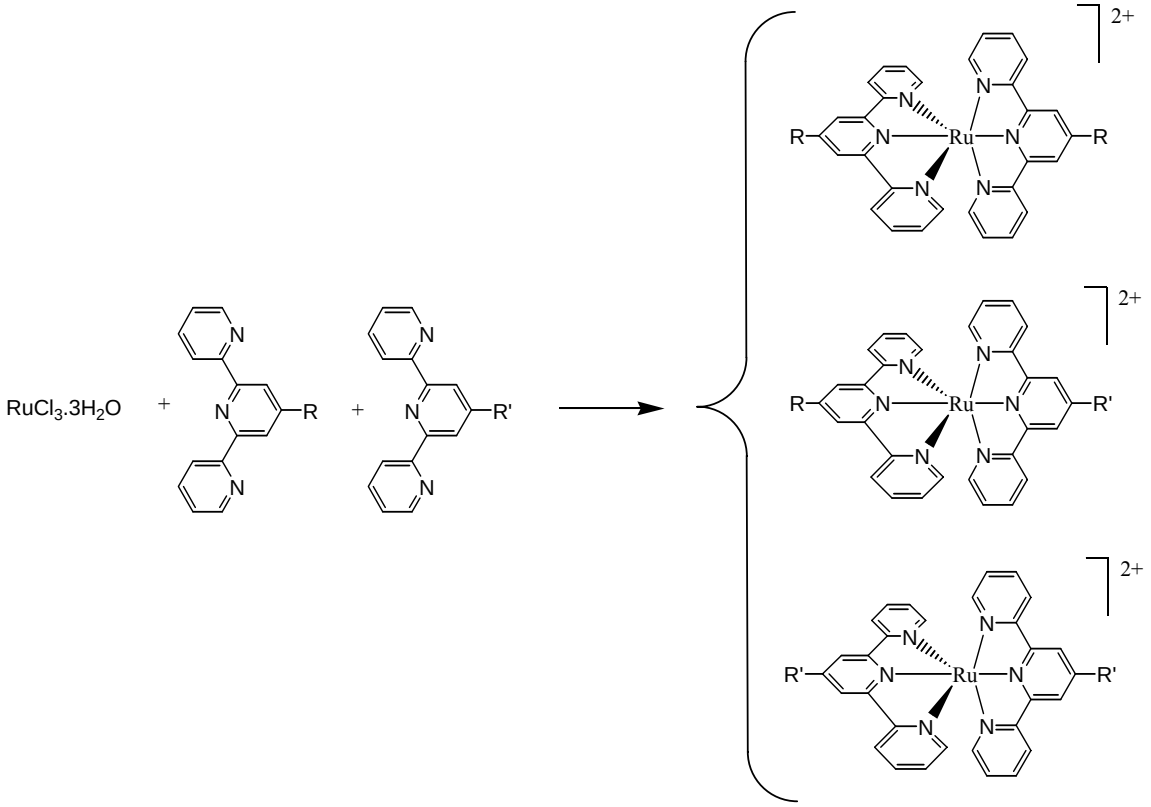
Scheme 1.22. (i) base (triethylamine or K_2CO_3), appropriate dry solvent, reflux, 48 hr.

1.7.1.3. Ruthenium Complexes of Terpyridine Systems

Ruthenium(II) polypyridine complexes have been the focus of considerable attention over the last few decades.[124,143,155,156,158,186,190,195,199,229,230]

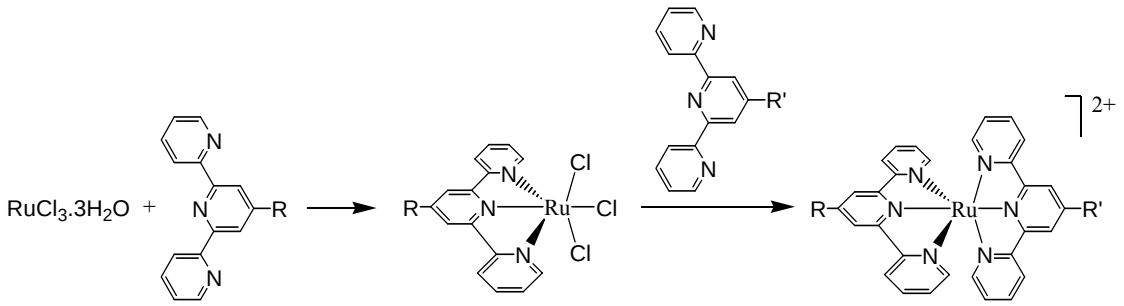
Terpyridine-ruthenium complexes of the type $[Ru(tpy)_2]X_2$ ($X = Cl^-$, ClO_4^-, PF_6^-) are already well-known.[122,231] A main characteristic is the strength of the metal-ligand bond in these complexes. With many transition metal ions in low oxidation states a bis-complex is formed, with pseudo octahedral coordination at the metal centre. The stability of this type of complex can be explained by the strong metal-ligand (d-π*) back donation. Furthermore, a strong chelate effect is present. The common geometry of this type of complex is a distorted octahedral geometry, since the most common coordination for transition metal ions is hexacoordination. This distorted octahedral coordination geometry has been characterised in detail by X-ray crystallography.[187,223,231-235]

In order to obtain bis(terpyridine) metal complexes, metal ions (*e.g.* zinc(II), cobalt(II), copper(II), nickel(II), iron(II) are usually treated with the respective ligand in a 2:1 ratio. The complexes are subsequently purified by exchange of the counterions, recrystallisation, or column chromatography of the complex. Addition of metal salts to a mixture of two terpyridines which are different in their 4'-positions, leads to a statistical mixture of homo and hetero complexes (Scheme 1.23).



Scheme 1.23

In order to prepare intentionally hetero complexes, step-wise reactions are introduced (Scheme 1.24).



Scheme 1.24

In the first step, ruthenium(III) trichloride hydrate was added to a methanolic or ethanolic solution of the first terpyridine ligand. The resulting mono-complex is

poorly soluble in most cases and can be easily isolated by filtration. Subsequently, this species is suspended with the second ligand in methanol, containing *N*-ethylmorpholine and refluxed for 1-4 hours. The solvent also acts as a mild reducing agent to reduce the ruthenium(III) to ruthenium(II), and also replace the chlorides to facilitate coordination of the second terpyridine.

Alternatively, an equimolar amount of $AgBF_4$ is added to the ruthenium(III) mono-complex in DMF or acetone to remove the coordinated chloride.[184,236,237] The vacant coordination sites are now occupied by the weakly binding solvent molecules, thereby activating the ruthenium(III) complex. Unlike terpyridine ruthenium(III) trichloride that is reacted in suspension, the activated species is soluble. After filtration of the AgCl precipitate, this intermediate is reacted without isolation with the second terpyridine ligand which leads to the desired heteroleptic ruthenium(II) complex. Yields between 50 % and 90 % can usually be obtained.[236,237]

Homoleptic ruthenium(II) complexes of terpyridine can be obtained either by using stepwise methodology or in a one-pot reaction.

1.6.1.4. Photophysical Properties of Polypyridine Ru(II) Complexes

Rich photophysical properties of polypyridine ruthenium complexes make them attractive candidates for applications as photosensitisers in light-harvesting devices (LHDs)[122,126,132,144,155,156,195,207,226,229,238-241] where they replace natural chromophores, such as chlorophyll-a and β-carotenoid in photosystem II (PSII). The ruthenium complexes absorb energy in the visible region of the spectrum giving rise to a singlet metal-to-ligand charge transfer (^{1}MLCT) excited state, which quickly

produces a potentially emitting triplet state (^{3}MLCT).[144,145,242] The ^{3}MLCT excited state may be sufficiently long-lived to transfer an electron or energy to a suitable acceptor depending on the complex under investigation.

The artificial counterparts for the chromophores of PSII have typically been based on the $[Ru(bpy)_3]^{2+}$ motif due to its relatively long-lived excited state at room temperature (1100 ns).[124,125] However, in larger polynuclear systems based on $[Ru(bpy)_3]^{2+}$, the stereogenic metal centres create diastereomers, and further substitution on the bpy ligands may lead to *facial* and *meridional* isomers.[122,203] Although methods to prepare enantiopure complexes based on $[[Ru(bpy)_3]^{2+}$ have been developed,[243] attention has turned to synthetically more accessible complexes based on tridentate ligands such as 2,2':6',2''-terpyridine (tpy), **1.3**.

$[Ru(tpy)_2]^{2+}$ has a relatively long luminescence lifetime in a rigid matrix at 77 K, however, at room temperature its excited state is quenched with a lifetime of only 250 ps.[208] The rigid tridentate ligands create a greater distortion from ideal octahedral geometry in their Ru(II) complexes.[17,123,124,127,132,142,143,145,147,156,158,184-188,190-193,195-200,202,206,210,229,232,237,240-242,244-255] Smaller N-Ru-N *trans* angles are found in coordinated tpy (158.6°) as compared to the analogues Ru(II) complexes with bpy (173.0°).[256,257] This gives rise to a weaker ligand field strength, which effectively reduces the energy of the dd metal-centred triplet state (^{3}MC) (Figure 1.26). A consequential decrease in the energy gap between the ^{3}MLCT and ^{3}MC is observed and the ^{3}MC becomes thermally accessible from the ^{3}MLCT, facilitating non-radiative decay back to the ground state (GS).[145]

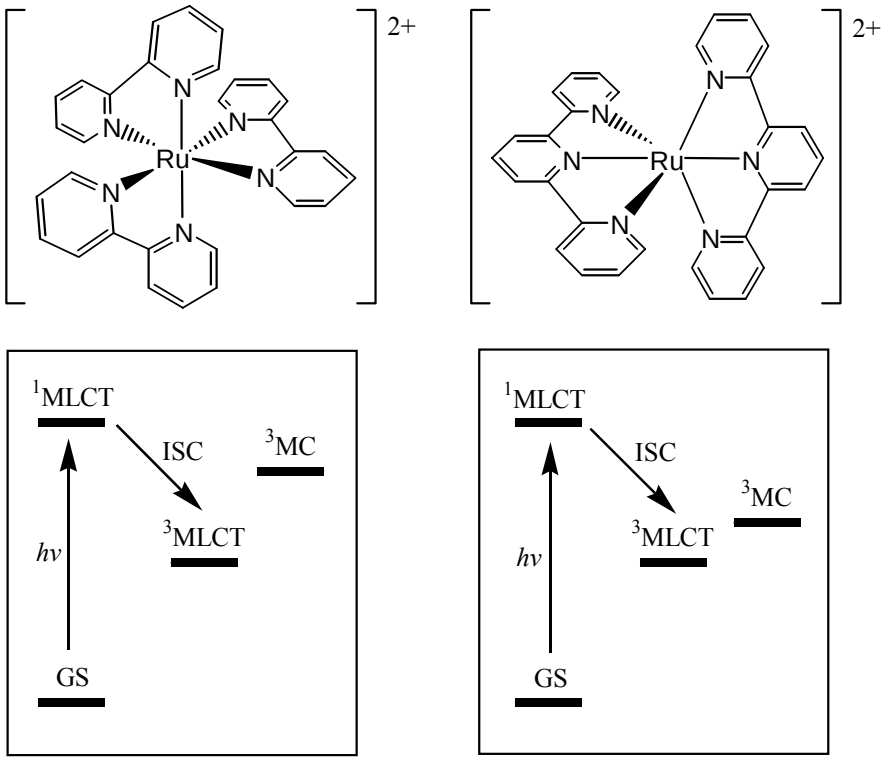


Figure 1.26. (a) Photophysically appealing $[Ru(bpy)_3]^{2+}$ versus (b) synthetically appealing $[Ru(tpy)_2]^{2+}$.[145]

The excited state lifetime of Ru(II) complexes is dependent on the radiative and non-radiative rate constants as given by:

$$\tau = \frac{1}{k^0 + k^{0'} \exp(-E_a/RT)} \qquad (1.1)$$

where $k° = k_r + k_{nr}$, the sum of the radiative and non-radiative rate constants, respectively, and $k°'$ relates to a thermally activated crossover process in which E_a is the activation energy barrier to the ^{3}MC state.[145,258] As Ru(II) polypyridine complexes are weakly emitting, the non-radiative decay constant is the more important factor of the two rate constants.[259] The two major pathways for non-radiative deactivation are through a direct contribution from the ^{3}MLCT state to the GS and through a thermally accessible ^{3}MC state back down to the ^{3}MLCT state. However, if the ^{3}MLCT state is too low in energy, the excited state lifetime may be shortened even more by the direct

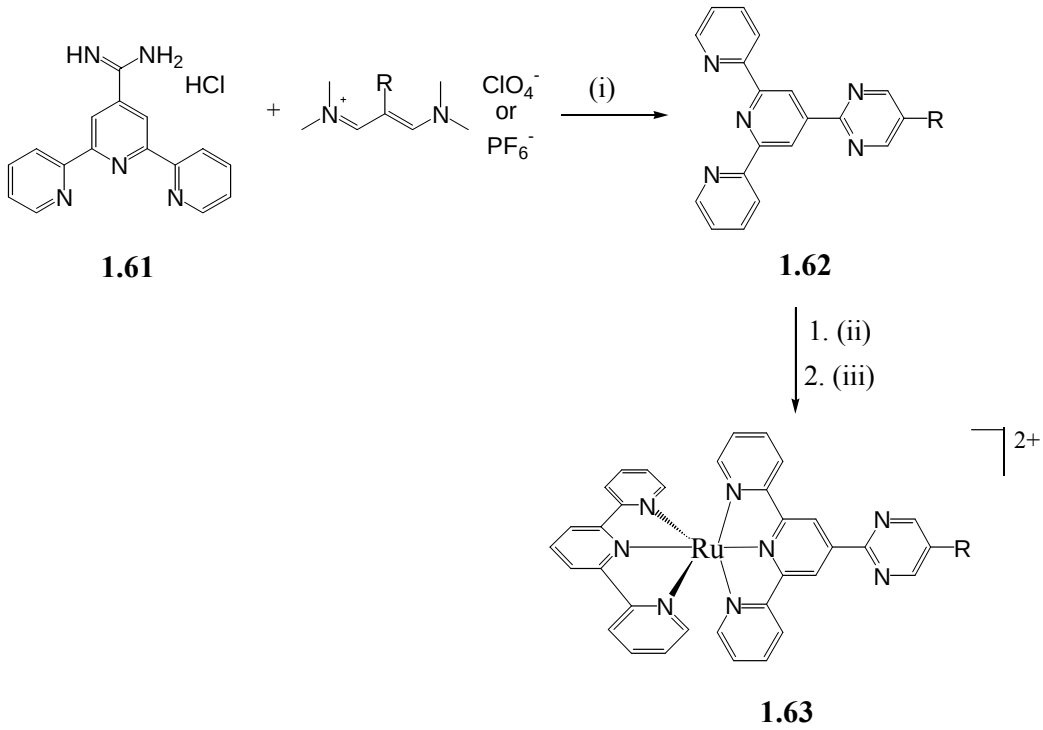
contribution back to the GS according to the energy gap law.[124,143,258,259] Although tridentate ligands based on the tpy motif have overcome the problems associated with the chirality of $[Ru(bpy)_3]^{2+}$, it has come at the expense of some of their excellent photophysical properties.

1.7.1.4. Enhancement of the Photophysical Properties of Ru(II) Complexes of Tridentate Ligands

The synthetic strategies used to enhance the photophysical properties of Ru(II) complexes of tridentate ligands have been studied by Hanan *et al.*[144-146,196,198,201,209,210,242,260] and also reviewed recently.[145] Hanan and his group have focused on manipulating the energy differences between the ^{3}MLCT and ^{3}MC states of the complex in order to minimise non-radiative decay through the ^{3}MC state or stabilisation of the ^{3}MLCT state, or both, leads to a greater energy gap between the two states. However, stabilisation of the ^{3}MLCT also reduces the energy gap to the ground state which is the usual deactivation pathway for low energy emitting Ru(II) complexes.

Four main approaches to enhance the photophysical properties of terpyridine-ruthenium complexes were considered:[144,145,196,201,209,210] (i) incorporation of an electron withdrawing or electron donating substituent onto tpy system; (ii) use of bichromophoric systems; (iii) increasing the energy of the ^{3}MC state; and (iv) π-electron accepting considerations.[145] Using bichromophoric systems, in which an organic chromophore with a non-emissive triplet state is similar in energy to an emissive ^{3}MLCT state and an equilibrium is established between the two states.[261] In order to have efficient energy equilibration in bichromophoric systems, complexes and

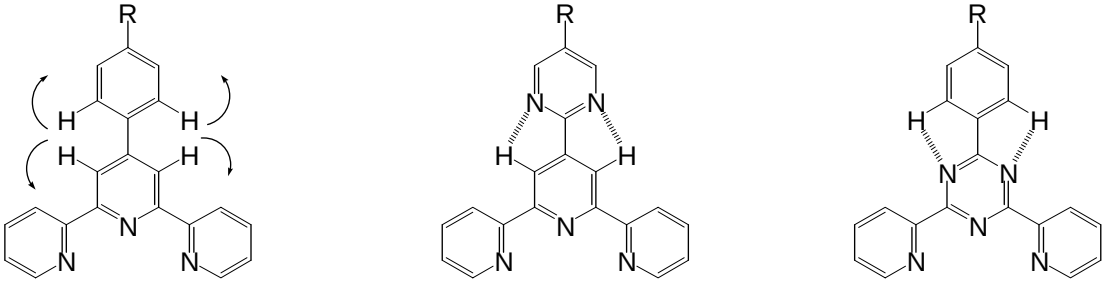
chromophores with isoenergetic triplet states are required. The synthetic strategies to prepare Ru(II) complexes containing pyrimidyl-terpyridine ligands can be used to fine-tune the ^{3}MLCT energy. (Scheme 1.25)



1.61 **1.62** **1.63**

Scheme 1.25. (i) MeONa, MeOH, reflux, 12hr; (ii) $Ru(tpy)Cl_3$, $AgNO_3$, EtOH; (iii) NH_4PF_6.

The energy of the ^{3}MC state can be possibly increased using σ-donors, cyclometallation, and decreasing the steric strain in the tridentate ligand.[145] To reduce the energy of the ^{1}MLCT state and consequently the ^{3}MLCT state, better π accepting ligands have been used. The central pyridyl ring of terpyridine can be replaced by a triazine ring while maintaining a N, N, N, coordination mode. Hanan *et al*[145] idea was based on the observation that appropriately substituted heterocyclic rings lead to a coplanar arrangement of rings, as opposed to the slight twist found between aromatic hydrocarbon rings (Figure 1.27).[260]



Non-planar Co-planar Co-planar

Figure. 1.27. Coplanar and nonplanar arrangements of some substituted tpy systems.

The incorporation of triazine rings and the near planarity of the rings arising from intramolecular H-bonding between the N atoms on the triazine ring with the hydrogen atoms on the pendant phenyl ring (Figure 1.27). Delocalisation of the rings from removal of steric clashes increases the luminescence life-times of the Ru(II) complexes by lowering the energy of the ^{3}MLCT state and creating a large energy gap to the dd state (also see Figure 1.26).[145,260]

1.7.2. Acceptor Site

A number of possibilities were considered for the second metal ion binding site (acceptor site, A). Table 1.1 shows some cyclic/acyclic polyamines, polypyrazolyl, and polypyridine binding domains as candidates for binding Co(III). These functional groups can be attached to the ttp ligand at its 4'-position.

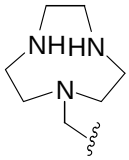
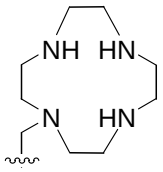
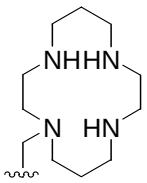
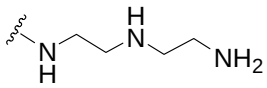
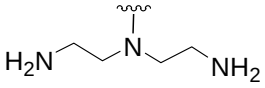
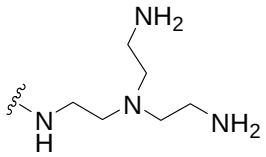
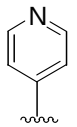
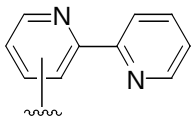
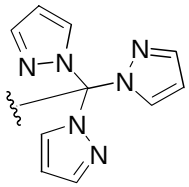
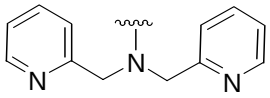
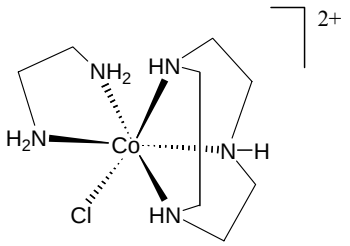
Macrocyclic polyamines	 -tacn	 -cyclen	 -cyclam
Chain Polyamines	 -1-dien	 -2-dien	 -tren
Aromatioc heterocyclic motifs	 -4-py	 -bpy	 -tpm	 -2-picolylamin

Table 1.1

We were again interested in reducing the number of stereoisomers that might be produced. In this context a facial tridentate binding site seemed attractive, as it offered the benefits of a polydentate ligand, a minimisation of isomeric possibilities, and scope for differentiating that binding site from the meridional terpyridine binding site that we proposed to use at the other end of the ligand. The tris(pyrazolyl)methane (tpm) and 1,4,7-triazacyclononane (tacn) fragments (Table 1.1) fit these criteria, and the coordination chemistry of such systems has also been studied.[262-287] [288-290] Figure 1.28 shows a complex with facial geometry around the cobalt ion which can be produced as a single isomer in the reaction mixture.[289,290]

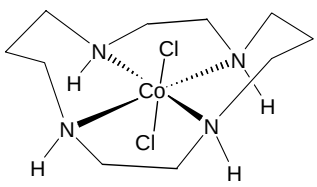


$[Co^{III}(tacn)(en)Cl]^{2+}$

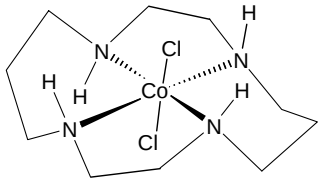
1.64

Figure 1.28. Structure of facial geometry of $[Co(tacn)(en)Cl]^{2+}$, **1.64**, complex.[289,290]

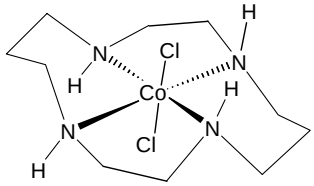
In 1,4,8,11-tetraazacyclotetradecane (cyclam) (see Table 1.1), however, each nitrogen atom, when coordinated, is an asymmetric centre, and five possible distinct non-enantiomeric combinations can be produced.[291] Some structures are shown in Figure 1.29 in which the *cis* isomer **1.69** can be separated and used for our research purposes.



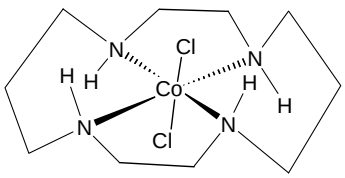
trans, **1.65**



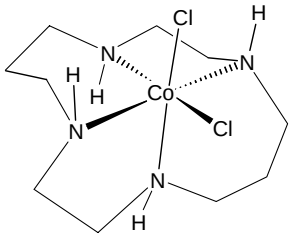
trans, **1.66**



trans, **1.67**



trans, **1.68**



cis, **1.69**

Figure 1.29. Some isomers of the strain-free octahedral Co(III) complexes of 1,4,8,11-tetraazacyclotetradecane (cyclam).

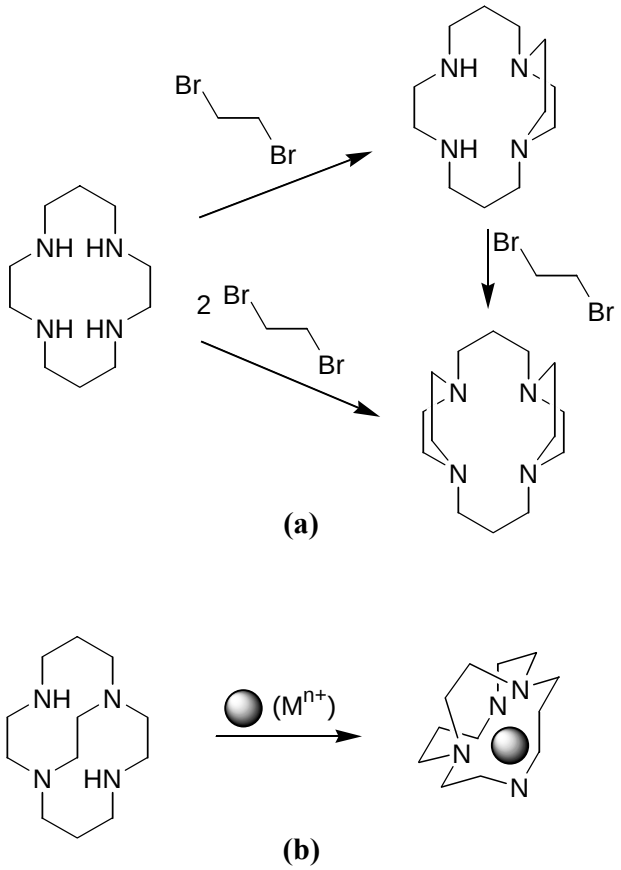
There are also some synthetic approaches available in the literature by which the number of possible stereoisomers of octahedral metal-cyclam complexes can be reduced. Some of these approaches are mentioned below.

1.7.2.1. "Bridged" and "Cross-bridged" Cyclam: Strategies to Reduce Possible Stereoisomers upon Complexation

Wainwright[292] observed that the interaction of 1,2-dibromoethane with cyclam results in the bridging of adjacent secondary amine donors by way of ethan-1,2-diyl bridges, thus providing a means for structurally reinforcing the backbone of saturated ligands without introducing unsaturation (Scheme 1.26 (a)). The introduction of such a bridge, or bridges, effectively creates a steric barrier, which molecular models strongly suggest as being sufficient to prohibit the possibility of ligand binding in anything other than the *trans* fashion.[292]

Weisman *et al*[293], however, investigated several members of ligands having nonadjacent nitrogen bridged by $(CH_2)_2$, the "cross-bridged" tetraamines including cyclam. They designed a bicycle[6.6.2] cyclam (Scheme 1.26 (b)) to be capable of adopting conformations, having all four nitrogen lone pairs convergent upon a cleft (*in,in* at the bridgehead nitrogens) for complexation.

Weisman *et al* also showed that the [6.6.2] cross-bridged cyclams indeed form *cis*-folded complexes with Cu(II) and Ni(II) ions.[293] They have also reported related Zn(II) complexes of cross-bridged cyclam demonstrating *cis*-folded complex.[294] No evidence for the presence of *trans* isomers has been found.

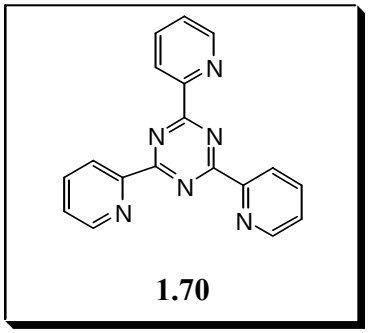


Scheme 1.26. (a) "structurally reinforced" cyclams to favour *trans*-coordination of transition metal ions.[292] **(b)** Metal complexation by "cross-bridged" cyclam.[295]

1.8. Thesis Coverage

In this thesis, as a whole, we describe synthesis and characterization of some terpyridine-based bridging ligands with two non-equivalent binding sites including some new ditopic ligands. Synthesis and characterization of some transition metal complexes of the prepared ligands are also described.

In Chapter 2, the ditopic bridging ligand 2,4,6-tris(2-pyridyl)-1,3,5-triazine (tpt), **1.70**, (Figure 1.30) is prepared and its coordination chemistry with Ni(II) is investigated. The paramagnetism of Ni(II) ion employed, dictate that X-ray crystallography be extensively used for the structural characterization of the products.

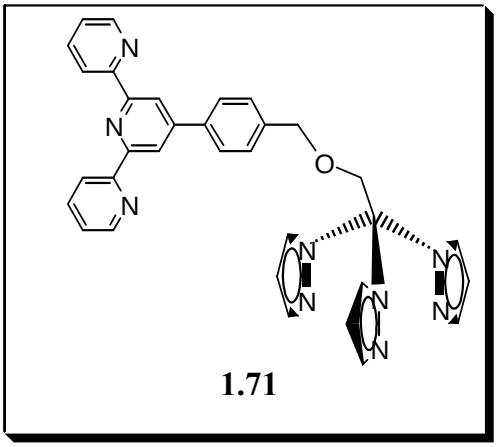


1.70

Figure 1.30. Ligand 2,4,6-tris(2-pyridyl)-1,3,5-triazine (tpt).

In Chapter 3, synthesis and characterisation of some polypyridine (ttp, bpy) and trispyrazolyl methane ligands are described.

In Chapter 4, a new ditopic ligand, 4'-(4-(2,2,2-tris(1*H*-pyrazol-1-yl)ethoxymethyl)phenyl)-2,2':6',2''-terpyridine (pzt), **1.71**, (Figure 1.31), has been prepared and its coordination chemistry studied. Metal ions (Fe(II) and Ru(II)) with a preference for octahedral geometry form ML_2 complexes that are readily isolated, with the metal ion being bound to the terpyridine sites of both ligands. Other metal ions (Ru(III), Cu(II), Zn(II), and Cd(II)) bind to the terpyridine site of just one ligand, and, in the case of silver(I), a dinuclear M_2L_2 complex has been isolated in which each silver ion is also coordinated to a single pyrazolyl donor group from the other ligand. Silver(I) ions react with FeL_2 and RuL_2 to create some coordination polymers incorporating Fe(II) and Ru(II) ions, respectively. Evidence for binding of metal ions to the tris(pyrazolyl) binding site was obtained by ESI-MS and NMR techniques. The free ligand and six metal complexes, including the disilver complex, have been characterised by X-ray crystallographic techniques.



1.71

Figure 1.31. A new ditopic ligand, 4'-(4-(2,2,2-tris(1*H*-pyrazol-1-yl)ethoxymethyl)phenyl)-2,2':6',2''-terpyridine (pzt).

In Chapter 5, preparation of some ditopic ligands containing polypyridines, cyclic and acyclic polyamines (Figure 1.32) were explained and synthesis of their ruthenium(II) complexes were also discussed. ESI-MS and NMR techniques were used to characterize the products. Subsequently, reactions of their ruthenium complexes with some transition metal ions including Co(III) ion are also described.

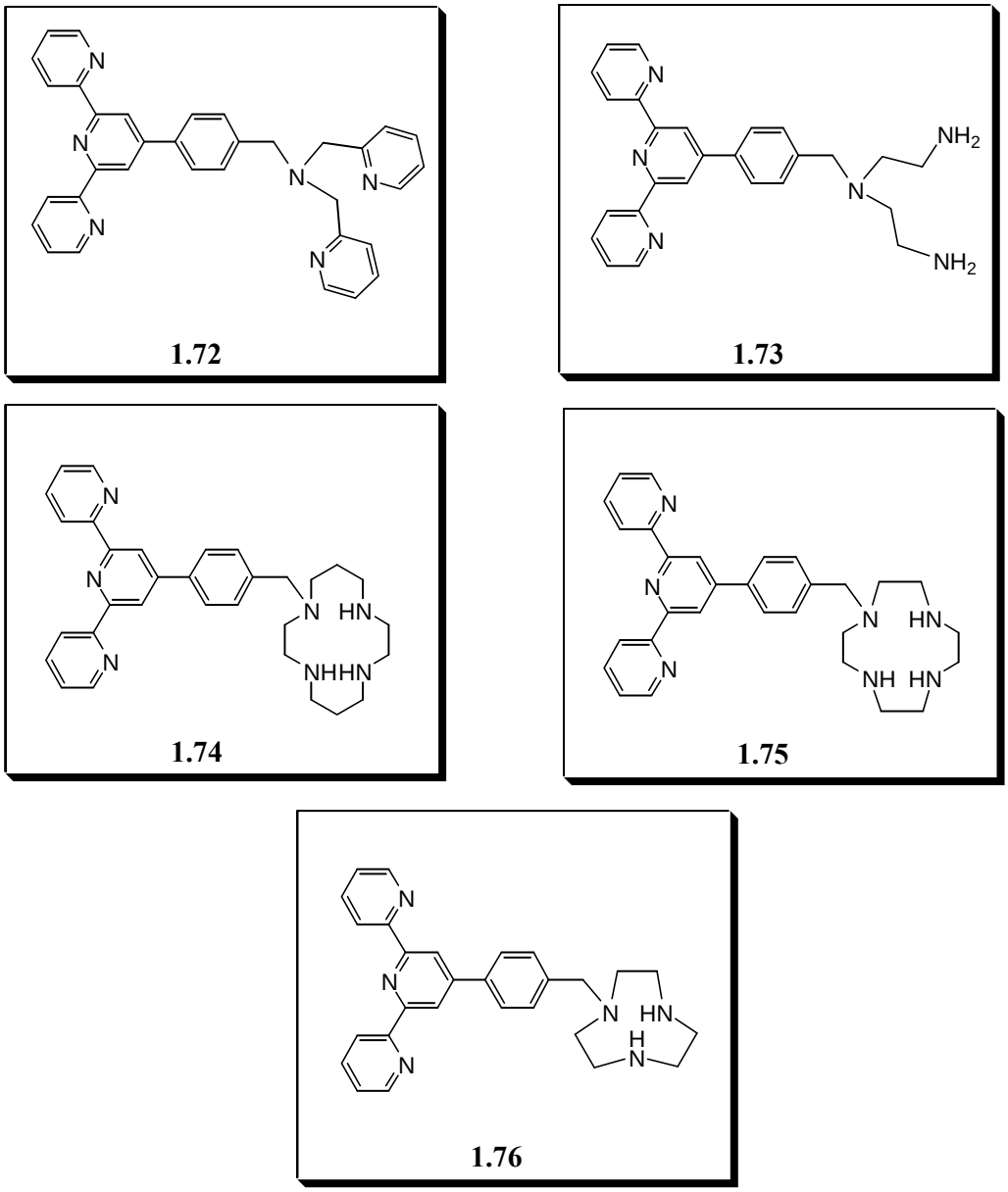


Figure 1.32. Some ditopic ligands with two non-equivalent binding sites.

In Chapter 6, structures of some incidental complexes are described.

In Chapter 7, conclusions and future prospects of this research are discussed.

In Chapter 8, the experimental work relevant to this thesis is described in detail.

The following articles based on work described in this thesis have been published:

1. Ramin Zibaseresht and Richard M. Hartshorn, *Aust. J. Chem*., **2005**, *58*, 345-353.

2. Richard M. Hartshorn, Ramin Zibaseresht, and Ward T. Robinson, *Acta Cryst*. **2005**. *E61*, m981-m983.

3. Ramin Zibaseresht and Richard M. Hartshorn, *Dalton Trans.*, **2005**, 3898-3908.

4. Richard M. Hartshorn and Ramin Zibaseresht, *ARKIVOC*, **2006** (iii), 104-126.

5. Ramin Zibaseresht and Richard M. Hartshorn, *Acta Cryst*. **2006**. *E62*, i19-i22.

6. Ramin Zibaseresht, Ward T. Robinson, and Richard M. Hartshorn, *Acta Cryst*. **2006**. *E62*, m1150-m1153.

Chapter 2

Synthesis and Characterisation of Some Ni(II) Complexes of 2,4,6-Tris(2-pyridyl)-1,3,5-triazine Ligand

2.1. Introduction

As was explained in Chapter 1, one of the most efficient ways to enhance the photophysical properties of terpyridine ligands is to replace the central pyridyl ring of terpyridine by a triazine ring while maintaining a tridentate coordination mode. The ligands 2,4-bis(2'-pyridyl)-6(4"-pyridyl)-1,3,5-triazine (bppt), **2.1**, and 2,4,6-tris(2-pyridyl)-1,3,5-triazine (tpt), **1.70,** (Figure 2.1) were interesting to us because they contain two non-equivalent binding sites: tridentate terpyridine-type "tail" binding site and a monodentate pyridine "head" binding site in **2.1**; or a bidentate bipyridine-type "side" binding site in **1.70**.

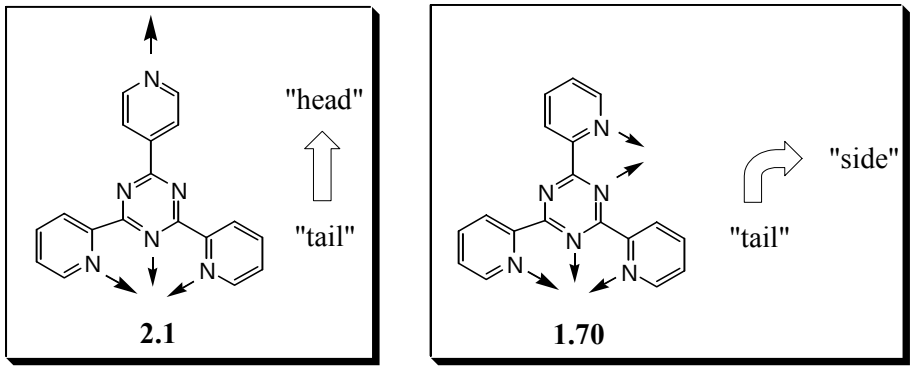
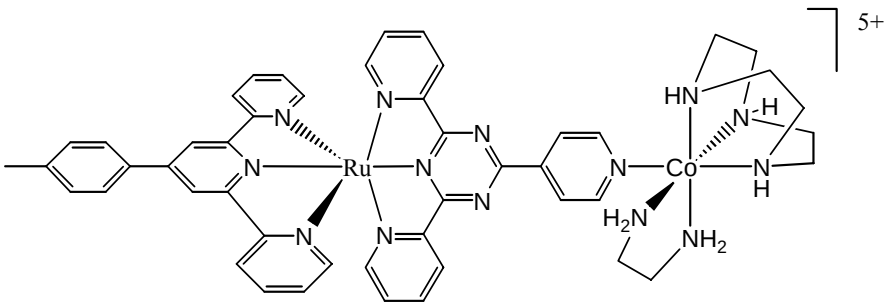
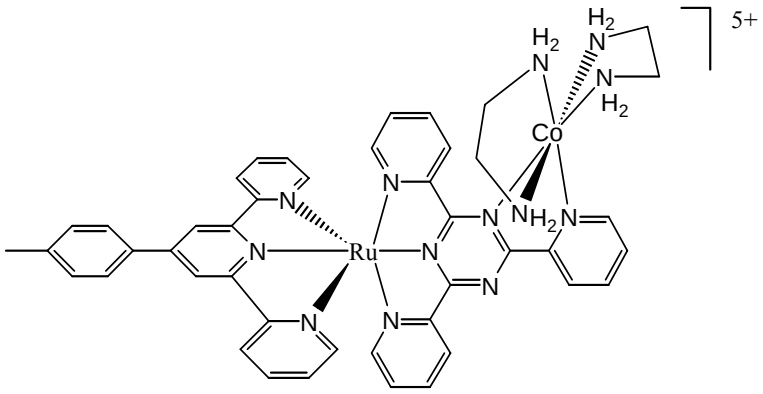


Figure. 2.1. The ligands 2,4-bis(2'-pyridyl)-6(4"-pyridyl)-1,3,5-triazine (bppt), **2.1**, and 2,4,6-tris(2-pyridyl)-1,3,5-triazine (tpt), **1.70,** with non-equivalent metal binding domains.

The ligands **2.1** and **1.70** are of interest; because they have the potential to behave as bridging ligands between two different transition metals (*e.g*. Ru(II) at the tridentate "tail" binding site, and Co(III) at the "side" or the "head" binding sites) (Figure 2.2). They are, therefore, candidate bridging ligands for use in the donor-acceptor systems required for our study.



2.2

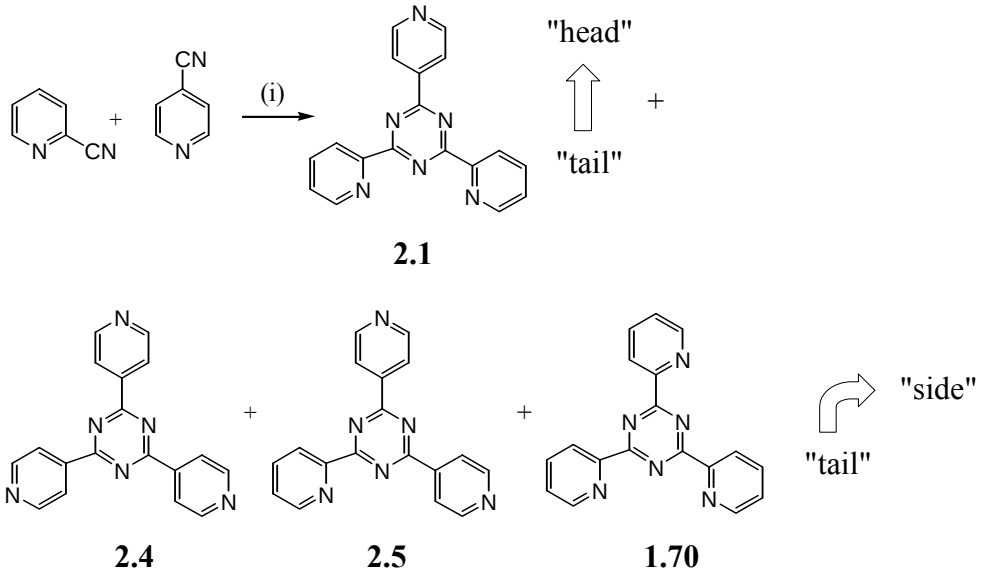


2.3

Figure 2.2. Potential target molecules **2.2** and **2.3** for HSC based-on (bppt) and (tpt), respectively as bridging ligands mediating a Ru(II) and a Co(III) metal centres.

2.2. Ligands Syntheses

Ligand **2.1,** along with several other compounds (**2.4**, **2.5**, and **2.6**) was produced from a melt reaction of 4-cyanopyridine and 2-cyanopyridine with NaH as catalyst.[209] (Scheme 2.1).



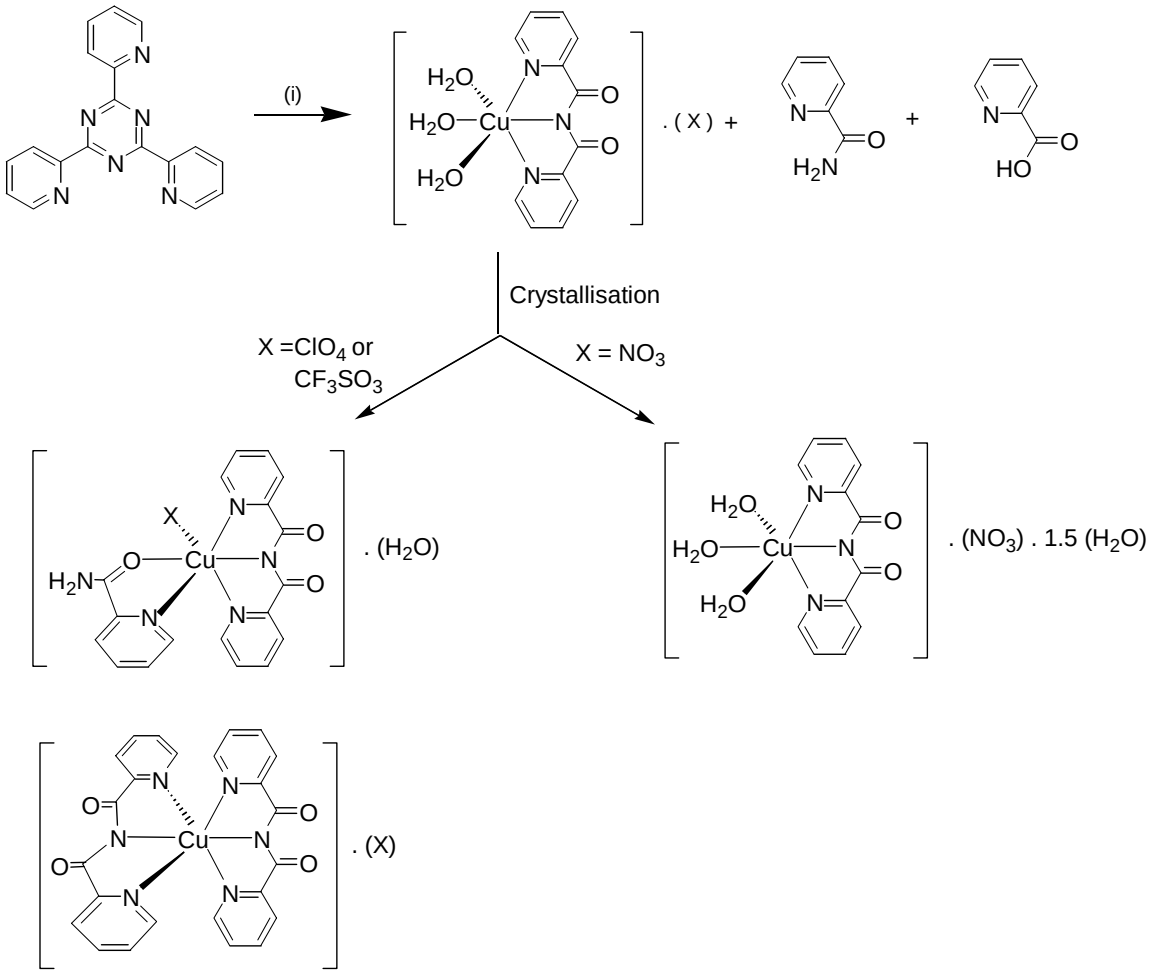
Scheme 2.1. Synthesis of 2,4-bis(2'-pyridyl)-6(4"-pyridyl)-1,3,5-triazine (bppt), **2.1**, as a major product in the reaction mixture. (i) NaH, 180 ºC.

This ligand offers the synthetic adaptability of terpyridine and the longer-lived excited-state lifetimes of bipyridine complexes. As such it represents an excellent candidate for use as a probe in photophysical studies of larger polymetallic systems.

The electrochemistry and the photophysical properties of ruthenium complexes of **2.1** are similar to that of $Ru(tpy)_2^{2+}$.[209]

According to Hanan *et al*,[209] the desired ligand could be separated from the mixture by a "complexation-decomplexation" strategy using Ni^{2+} ion as a complexation agent and KCN as a ligand liberator.

Ligand **1.70** is of current interest because of its use as a spacer for designing multinuclear metal complexes.[186,296-299] The ligand **1.70** has been widely used as an analytical reagent for various metal ions.[300-305] A number of transition-metal and lanthanide complexes of it have also been reported.[306-325] It is usually stable towards hydrolysis, concentrated mineral acid, and temperatures above 150 ºC are required for its hydrolytic reaction.[326] However, Lerner and Lippard[327,328] first found that **1.70** and a similar compound **2.1** undergo hydrolytic reaction in the presence of Cu(II) in aqueous media (Scheme 2.2), resulting in the formation of bis(2-pyridylcarbonyl)amido anions, which remain coordinated to Cu^{2+}, free pyridine-2-carboxamide, and free pyridine-2-carboxylic acid.[329,330]



Scheme 2.2. Hydrolysis of the ligand **1.70** in presence of copper ions in water.

Crystal structures of copper(II) complexes of the hydrolytic products of **2.1** and **1.70** have also been reported. [327,328,330,331] Figure 2.3 shows the view of the structure of $[Cu((NC_5H_4CO)_2N)(NC_5H_4CONH_2)(CF_3SO_3)]$ complex.[327,328,330,331]

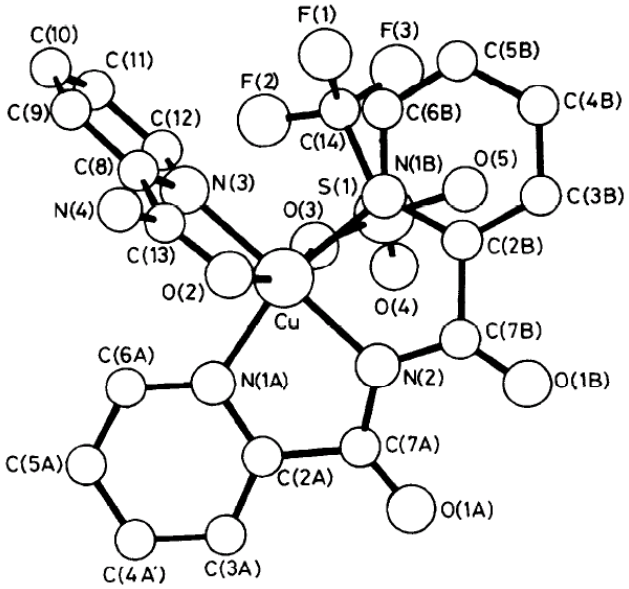


Figure 2.3. View of the structure of $[Cu((NC_5H_4CO)_2N)(NC_5H_4CONH_2)(CF_3SO_3)]$ complex. Hydrogen atoms are not included.

The hydrolytic reaction can be inhibited when N-donor tridentate ligands are bound to Cu^{2+} as illustrated by the determination of the structure of [bis(2-pyridylcarbonyl)amido][2,4,6-tris(2-pyridyl)-1,3,5-terpyridine]copper(II) trifluoromethansulfonate.[331]

The similar hydrolysis reaction was also observed upon reacting $RhCl_3 \cdot 3H_2O$ with **2.6** in refluxing ethanol-water.[332,333]

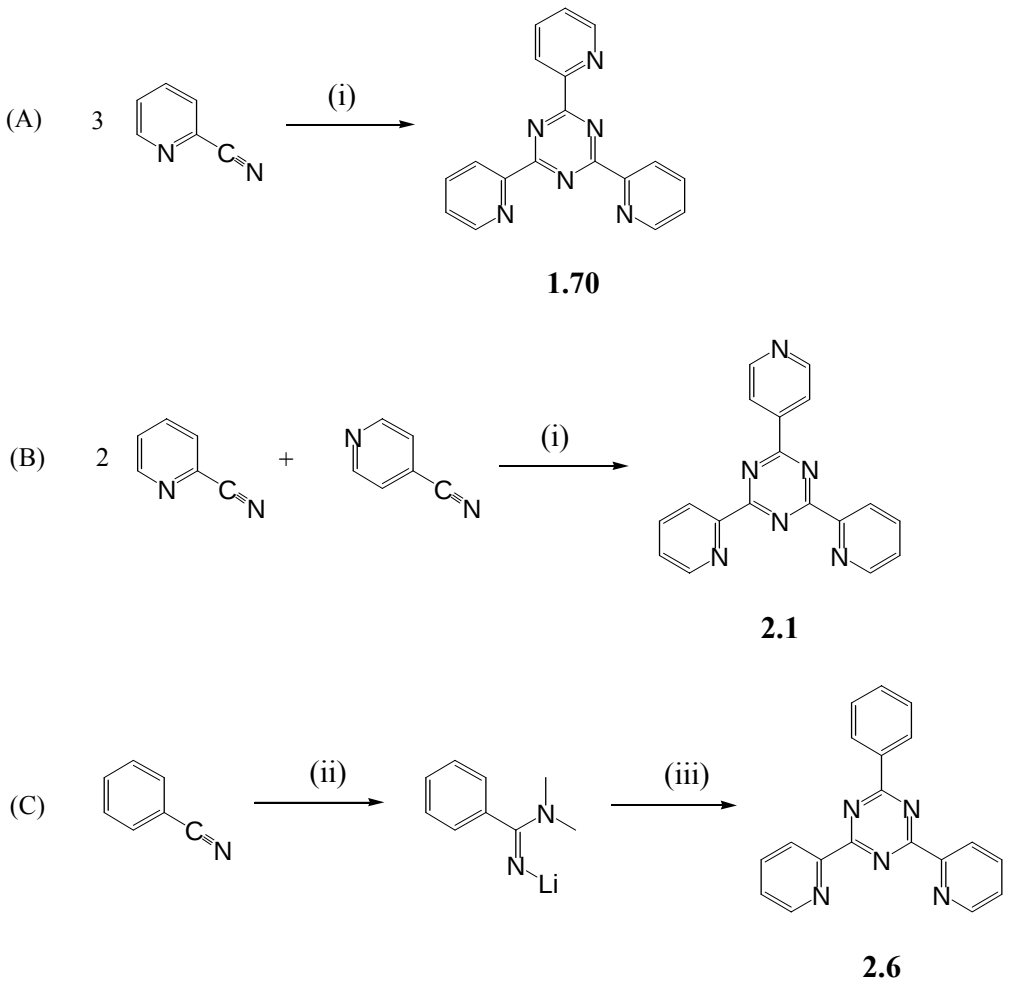
According to D. A. Durham *et al*, when **1.70** functions as a tridentate ligand, the coordination of a second metal to **1.70** is not usually observed due to deactivation of the triazine ring by the inductive effect of the first metal and as a result of steric interactions between the hydrogen atoms and the metal ion.[309] It has been also reported

that this versatile ligand may also function as a bis-bidentate ligand.[296,311,314,334,335] The coordination of two low-valent metal ions to **1.70** leads to destabilization of the triazine ring by enhancing its electron deficiency; nucleophilic reaction may thus occur at the carbon atom on the triazine ring which is adjacent to uncoordinated pyridyl ring.[336]

Several literature examples, however, demonstrate that the triazine ligand **1.70** is indeed able to coordinate a metal ion in both tridentate and bidentate sites.[297,311,316,337-339]

We have also found that during the purification of the crude reaction mixture, ligand **1.70** was also able to complex to two Ni atoms at its tridentate "tail" binding site as well as its bidentate "side" binding site, simultaneously. The synthesis of the target molecule shown in Figure 2.2 may, therefore, be feasible.

An efficient synthetic methodology has been developed for a range of triazine-based ligands. Ligand **1.70** was prepared by trimerisation of 2-cyanopyridine in the presence of a catalytic amount of NaH[209,340] (see Scheme 2.3). The ligands with aromatic hydrocarbon rings such as **2.1** were synthesised by treating two equivalents of 2-cyanopyridine with the lithium amidinide salt of the appropriate aromatic hydrocarbon in diethyl ether (Scheme 2.3).[209,260]



Scheme 2.3. Preparation of triazine-based ligands.[209,260] (i) NaH (catalytic amount), 180º; (ii) $LiNMe_2$, Et_2O; (iii) 2-cyanopyridine (2 equimolar).

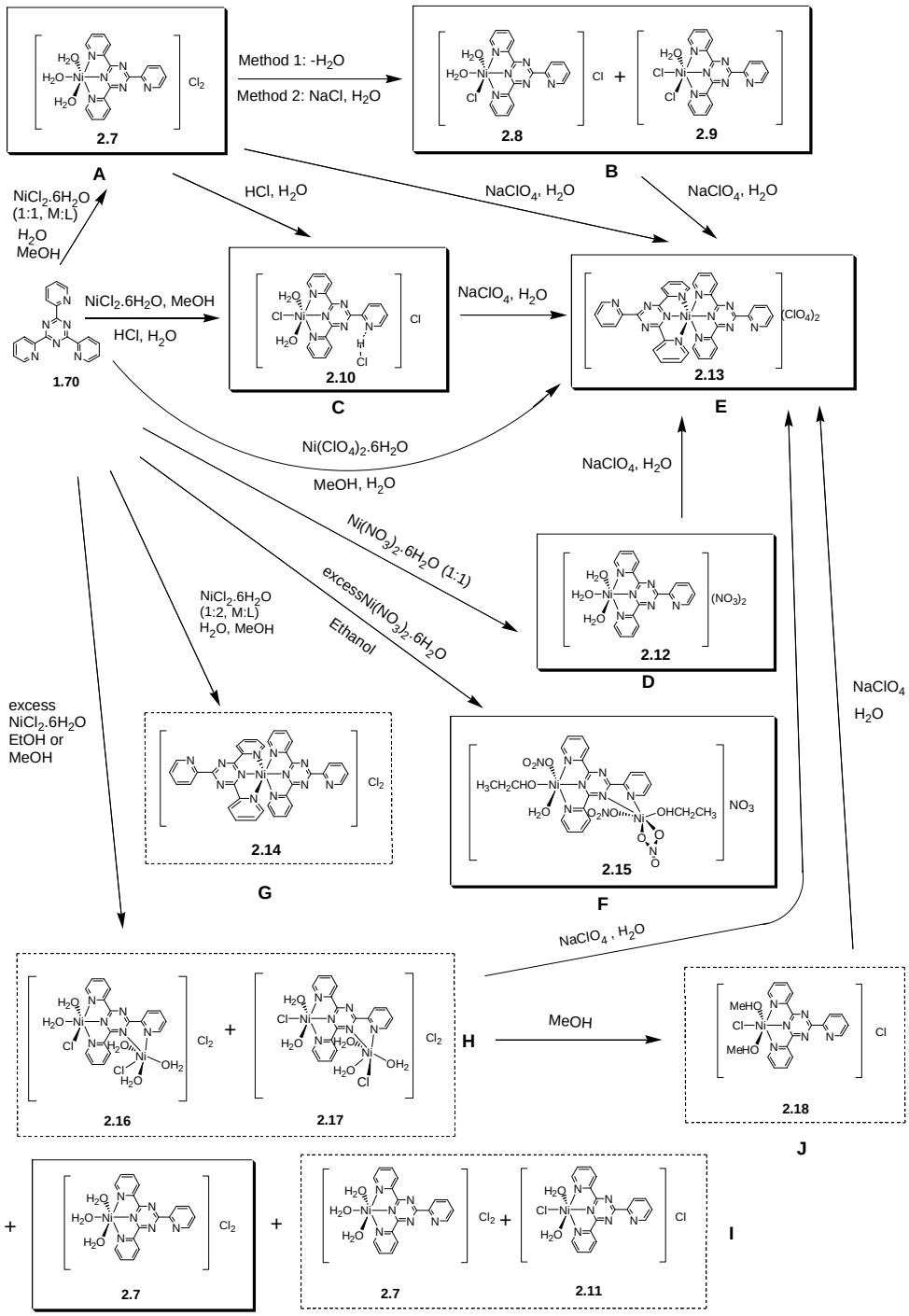
A series of nickel(II) complexes of ligand **1.70** have been prepared and identified using crystallographic techniques and will be discussed in this chapter.

The triazine ligand **1.70** appealed to us initially, both because of the potential synthetic utility of the tridentate-bidentate binding site combination (which might allow sequential coordination of different metal ion synthons), and because of the ease with which the ligand can be synthesised.[209,340]

The potential versatility of this ligand has been recognised by others,[320] and the nickel(II) coordination chemistry of the ligand was explored some time

ago.[317,319,320,341] As will also be described later in this thesis (Chapter 6), preparation of dinuclear Ru(II)-Co(III) complexes of the tpt system to obtain a target molecule **2.3**, for example, was attempted. Unfortunately, our attempts to synthesise such heterodinuclear systems were unsuccessful, although no hydrolysis of the mononuclear Ru(II) complexes containing tpt ligand was observed. The stability of tpt ligand towards hydrolysis was also supported by others.[186,209,260,335] To the best of our knowledge, there is no report on Co(III) complexes of tpt ligands in the literature, but mono- and di-nuclear Co(II) complexes of this ligand were synthesised and described in the literature.[308,314-316,334,342-347] Our interest, however, in the coordination chemistry of this ligand led us to revisit some of this chemistry, with the intention of applying modern crystallographic and spectroscopic techniques to the system. In particular, we were interested in characterising the coordination modes present in the dinickel-tpt complex that had been described.

Crystalline material was obtained for a dinuclear complex and an X-ray structure determination was attempted. Unfortunately, the refinement of the structure solution for those crystals proved somewhat problematic, and ultimately gave only a poor structure solution. The results of the study were, however, consistent with the proposed dinuclear coordination mode. Furthermore, the search for conditions under which better quality crystals of the dinuclear complex might be formed led to other complexes being isolated and, as we explored this chemistry further, a series of related complexes was prepared. This chapter outlines the conditions required to produce a range of nickel complexes of ligand **1.70**, and details of the structures of the crystalline compounds that we isolated. A total of seven nickel complexes of ligand **1.70** have been isolated and crystallographically characterised in this work, while a further six have been tentatively identified (Scheme 2.4).



Scheme 2.4. [348] Syntheses of nickel(II)-tpt complexes. The molecules indicated in the solid boxes have been crystallographically characterised and fully refined. Complexes inside the same box co-crystallised. The molecules shown in the dotted boxes were crystallographically characterised, but the refinements were poor or incomplete, so the structural identification of these latter complexes remains tentative.

2.3. Results and Discussion

The synthetic chemistry that links the materials and complexes described in this chapter is presented in Scheme 2.4. A series of 1:1 nickel-tpt complexes have been prepared, sometimes as mixtures, and under some conditions, 1:2 and 2:1 complexes can also be isolated. All of the compounds in the scheme have been isolated in crystalline form, and their structures studied using X-ray crystallographic techniques. Satisfactory refinements were achieved for the compounds in solid boxes, whereas only poor refinements resulted from data collected for the remaining compounds (in the dotted boxes). We believe the refinements in the latter cases are sufficiently good to at least identify the compounds, but we note that these identifications should be regarded as tentative. Detailed descriptions of the infrared spectra of these compounds are provided in the experimental section of this thesis, so that these materials can be identified by other workers without resort to diffraction techniques.

The results of the crystallographic studies on the nickel-tpt complexes reveal that the metal centres are approximately octahedral, with three coordination sites occupied by each tpt ligand, **1.70**, (except in the dinuclear systems where the second metal ion has only two sites occupied by the tpt ligand). In most cases, the remaining coordination sites on the nickel ions are filled by a mixture of chloride and water ligands, and different compounds and stereoisomers are found, occasionally within the same crystals.

Tridentate coordination of ligand **1.70** to the nickel ion in these structures results in bonds to the central triazine nitrogen donor that are significantly shorter than those to the two pyridyl nitrogen donors (Table 2.1). This observation is consistent with the vast majority of the literature data for metal complexes containing the di-2-pyridyltriazine fragment,[297,301,311,316,319,320,322,331-333,337-339,341,345,349-353] the exceptions

that we are aware of being a tin complex,[354] a praseodymium complex,[352] and two lead complexes,[339,350,355] where one of the bonds to a pyridine donor is shorter than the bond length to the triazine nitrogen donor. The shorter bond to the central donor is also seen for nickel(II) complexes of ligands containing terpy-type fragments.[113,187,235,356-384] By contrast, bidentate coordination of a triazine nitrogen donor and just one pyridyl nitrogen donor results in a longer bond to the triazine ring than to the pyridine ring, and again this is consistent with the literature where Ru(II), Hg(II), Co(II), and Cu(II) ions were bound to the bidentate site of the tpt ligand.[297,311,316,337-339]

Complex / Ni-N	Complex **2.7**	Complex **2.8**	Complex **2.9**	Complex **2.10**	Complex **2.12**	Complex **2.13**	Complex **2.15**
Ni-N(Ct)*	1.977(4)	1.987(2)	1.989(2)	2.0126(16)	1.993(2)	2.053(3), 2.054(2)	1.969(3)
Ni-N(Sd)#	2.162(4)	2.171(2)	2.144(2)	2.1566(17)	2.148(2)	2.225(3), 2.211(3)	2.118(3)
Ni-N(Sd)#	2.160(4)	2.151(2)	2.145(2)	2.1778(17)	2.162(2)	2.220(3), 2.216(3)	2.110(3)

Table 2.1. Selected bond lengths [Å] for the Ni-tpt complexes.* Central triazine pyridine ring; # Side pyridine rings.

Once the isolated compounds have been characterised, the ligand sets that are found can be rationalised on the basis of the reaction conditions, but it does not appear possible to predict the exact results of using a particular set of reaction conditions. Some general principles can be used to at least predict the kinds of molecules that will be found, however. For example, a 1:1 nickel-tpt ratio, in water, will usually produce the tris(aqua) complex, but if the concentration of a (weakly) coordinating anion (*e.g.*

chloride, nitrate) is increased, either by evaporation of the solvent or addition of more anions, then complexes with coordinated anions will be isolated. Higher concentrations of anions will result in more of them displacing water ligands. Exactly which compounds will be isolated presumably depends on the exact reaction conditions, the relative amounts of the various species (including stereoisomers) that are present in solution, and the solubility of the various possible crystalline materials. Clearly, the number of possible crystalline materials that would have to be considered becomes rather large when, as we have observed, different combinations of complexes can co-crystallise. Use of alcohol solvent can also result in displacement of the water ligands.

Dinickel complexes of the kind that were of most interest to us are formed and can be isolated from the reaction mixture only in the presence of excess metal ion. The incomplete refinement of Structure **H** was our first evidence for the formation of these complexes, and the structure that was obtained also alerted us to the possibility that different stereoisomers may be found within the same crystal in these systems. Subsequently conditions were found that resulted in our determining the structure of compound **2.15**, a dinickel complex containing nitrate and ethanol ligands (Scheme 2.4). The structure is also shown later in Section 2.4.

The approximately octahedral nickel ions in this structure are coordinated by a variety of ligands in addition to the tridentate and bidentate binding site donor atoms of the ligand **1.70**. The nickel ion in the tridentate site also carries an ethanol ligand, a monodentate nitrate ligand, and a water ligand. The other nickel ion is coordinated by two nitrate ions, one monodentate and one bidentate, as well as an ethanol ligand. There is an extensive hydrogen bonding network that involves the non-tpt ligands and lattice-bound water molecules (Figure 2.4). There are also π-π stacking interactions

(face-face, 3.6 Å (centroid-centroid)) between the side rings of adjacent dinuclear cations in the lattice.

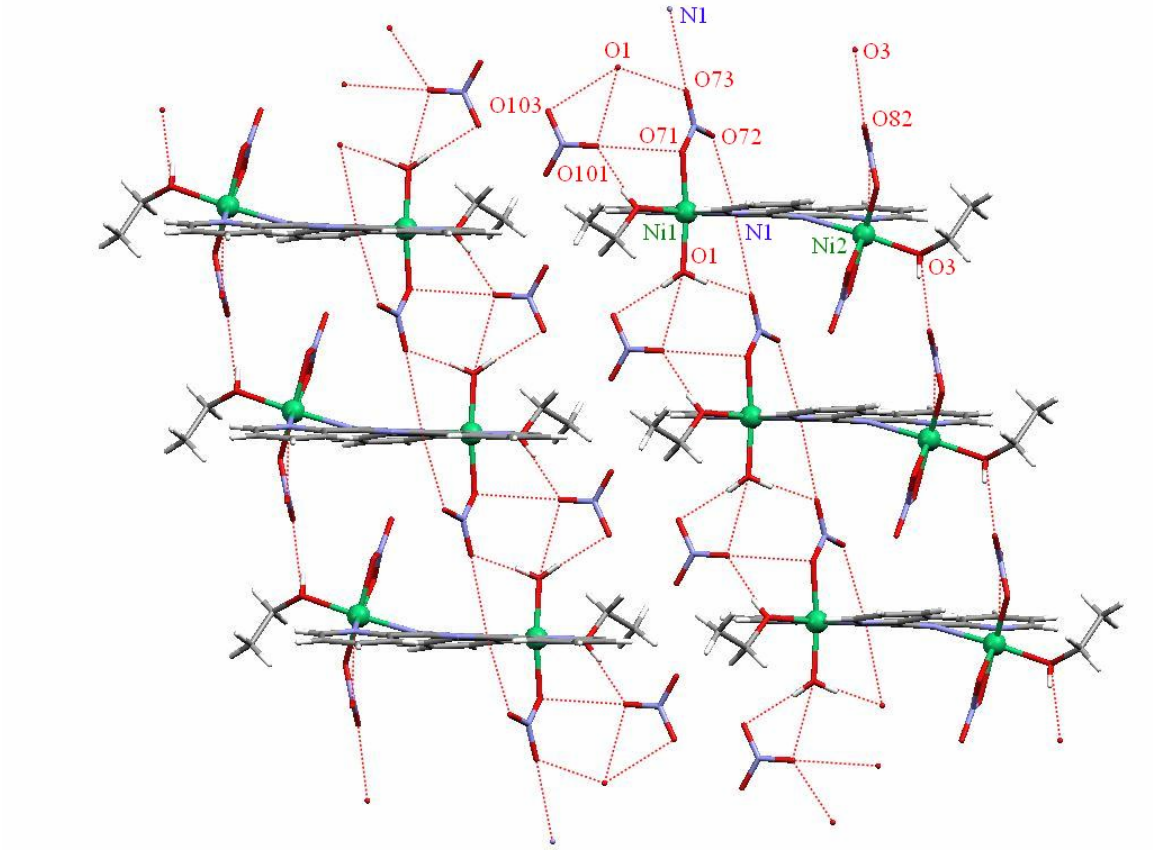


Figure 2.4. Hydrogen bonding network in the lattice of complex **2.15** (Structure **F**).

If a significant excess of nickel was not present, 1:1 nickel-tpt complexes were isolated, and the 1:1 compounds were also found if there is water present in the reaction mixture. Presumably negatively charged ligands stabilise complexes where two metal ions are bound to the triazine ligand, but the apparent preference of the nickel ions for water ligands rather than chloride or nitrate precludes this stabilisation in the aqueous systems that we used.

Several literature examples demonstrate that this triazine ligand is indeed able to coordinate a metal ion in both tridentate and bidentate sites.[297,311,316,337-339] The

conditions of the preparation of the dicopper[337] and dicobalt[316] complexes are consistent with the findings in this thesis in that the dinuclear species is produced when a coordinating anion (chloride or nitrate) is used in a non-aqueous system (methanol or ethanol). The Ru-Ag complex[297] makes use of only a single equivalent of silver ions, but in this case the silver ion carries only a single charge (so overall charge on the complex is less of an issue). The silver ion is also a much softer metal ion, and may actually prefer the pyridine-type donors to the other potential ligands in the system.

In the presence of excess triazine ligand the bis-tpt complexes are isolated from the reaction mixture, and the addition of perchlorate ions leads to this complex being formed even in those cases where less than two equivalents of ligand are available (Scheme 2.4). The X-ray structure of this complex is also shown later in Section 2.4 (Figure 2.13).

The lability of the mono-tpt nickel complexes and the relative stability of the bis-tpt perchlorate salt are demonstrated by the fact that this species is isolated when any of the other compounds are dissolved and the solution then treated with perchlorate. The FTIR spectra of the solid materials from these reactions were identical to that obtained from the bis-tpt material that was crystallographically characterised. Furthermore, bis-tpt complex ions are observed in the electrospray ionisation mass spectra of all of the nickel-tpt complexes that are described in this chapter – often they are the dominant signal, either at a mass to charge ratio of 341 units, corresponding to the $[Ni(tpt)_2]^{2+}$ ion, or, in the presence of nitrate (compounds **2.12** and **2.15**), at 744 units, corresponding to the $[Ni(tpt)_2](NO_3)^+$ ion.

The isolation of the bis-tpt complex in the presence of perchlorate ions, even when a 1:1 Ni-tpt ratio is employed, may be a result of the complex being inherently

more stable than the other possible complexes, its very low solubility in water, or a combination of these factors. Clearly the isolation of crystalline material from labile systems will depend on the relative solubilities of the various species present, and the observed low solubility of the bis-tpt complex is likely to be a key factor leading to its formation under these conditions. However, the electrospray mass spectrometry data reveal that even in the solution phase the equilibria result in a significant amount of the bis-tpt complex being formed. This implies, at least, that none of the other complexes are significantly more stable than the bis-tpt complex, or otherwise one of them would have been found in the mass spectra.

Given that the nature of the isolated material will depend on the stability of the complexes in the solid state, it is worthwhile examining the crystal structure of each complex that was isolated with a view to identifying stabilising interactions that might contribute to the formation and/or crystallisation of that complex in preference to others. However, such an analysis must be tempered by the observation that although it is the bis-tpt perchlorate salt that appears to be one of the more stable species isolated during this project, there are no obvious π-π stacking or hydrogen bonding interactions revealed in its X-ray structure. Presumably the low solubility of the complex is related to the lack of hydrogen bonding, as the ability of a complex to be a hydrogen bond donor or acceptor in the solid state would presumably also correlate with the strength of its interactions with solvent water. This highlights the fact that the presence of stabilising hydrogen bonding interactions in crystal structures is a two-edged sword when it is being considered in relation to solubility in hydrogen bonding solvents such as water or alcohols. They may stabilise the solid state structure but their presence also means that stabilising interactions may be possible with solvent.

The chloride salt of the triaqua complex **2.7**, (Scheme 2.4), also shown later in Section 2.4 (Figure 2.7), is found to have a bond length to the water molecule bound *trans* to the triazine that is longer than those that are *cis*. At first glance this might be attributed to *trans* influence because of the short bond length to the triazine donor atom. However, there are other structures described in this chapter where the short bond to the triazine donor is observed but the *trans* donor actually has a shorter bond length than a similar ligand that is bound *cis* to the triazine. Particular counter examples include both complexes in Structure **B** (see Scheme 2.4), where there are either two water or two chloride ligands that are *cis* and *trans* to the triazine donor, and in both cases the bond *trans* to the triazine donor is shorter than that which is *cis*. The triaqua nitrate salt in the literature structure[341] and in Structure **D** are further counter examples. There is also a number of complexes from the literature where the *trans* ligand has a shorter bond. Complex **2.10** would appear to be the exception in having the long bond *trans* to the triazine donor atom.

A total of three crystal structures of the triaqua complex are now available for comparison: a previously reported nitrate salt,[341] space group *Cc*; the nitrate salt in Structure **D**, space group $P\bar{1}$; and the chloride salt (Structure **A**). In all three cases, the structures appear to be stabilised by strong hydrogen bonding networks, with the significant difference being that π-π interactions are found in both Structures **A** and **D** but not in the literature structure. The plane-to-plane distances in these structures are 3.5 and 3.6 Å (centroid-centroid), respectively, and the extent of the π-π interactions, shown in Figure 2.5, is particularly striking in structure **A**. Figure 2.6 illustrates the combination of π-π interactions and hydrogen bonding networks that is found in Structure **D**.

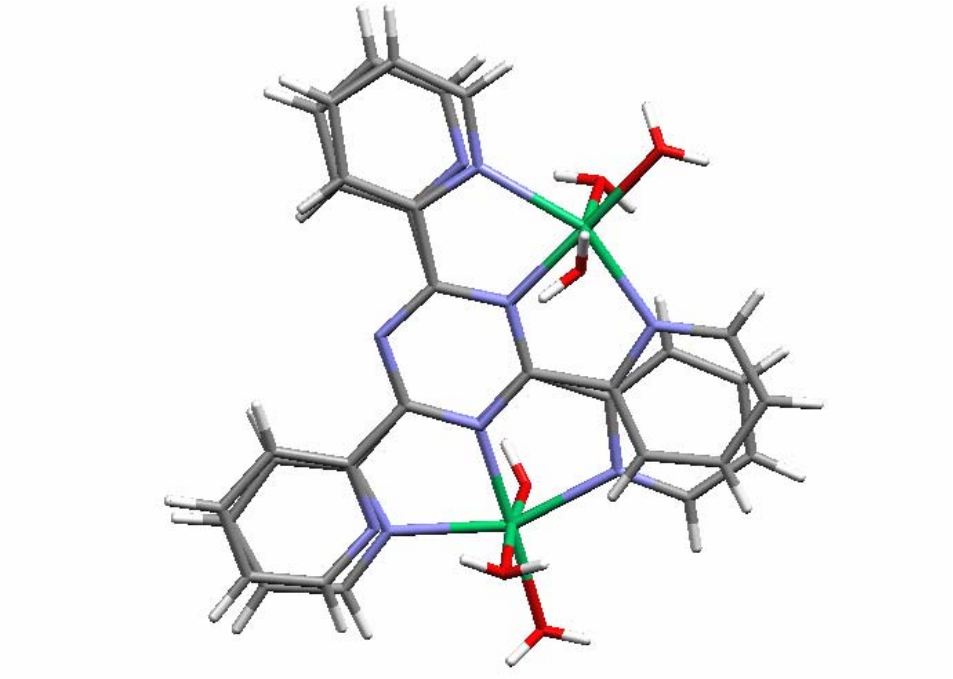


Figure 2.5. The π-π stacking interaction between two dications in Structure **A**. The distance between the central triazine rings are 3.5 Å (centroid-centroid).

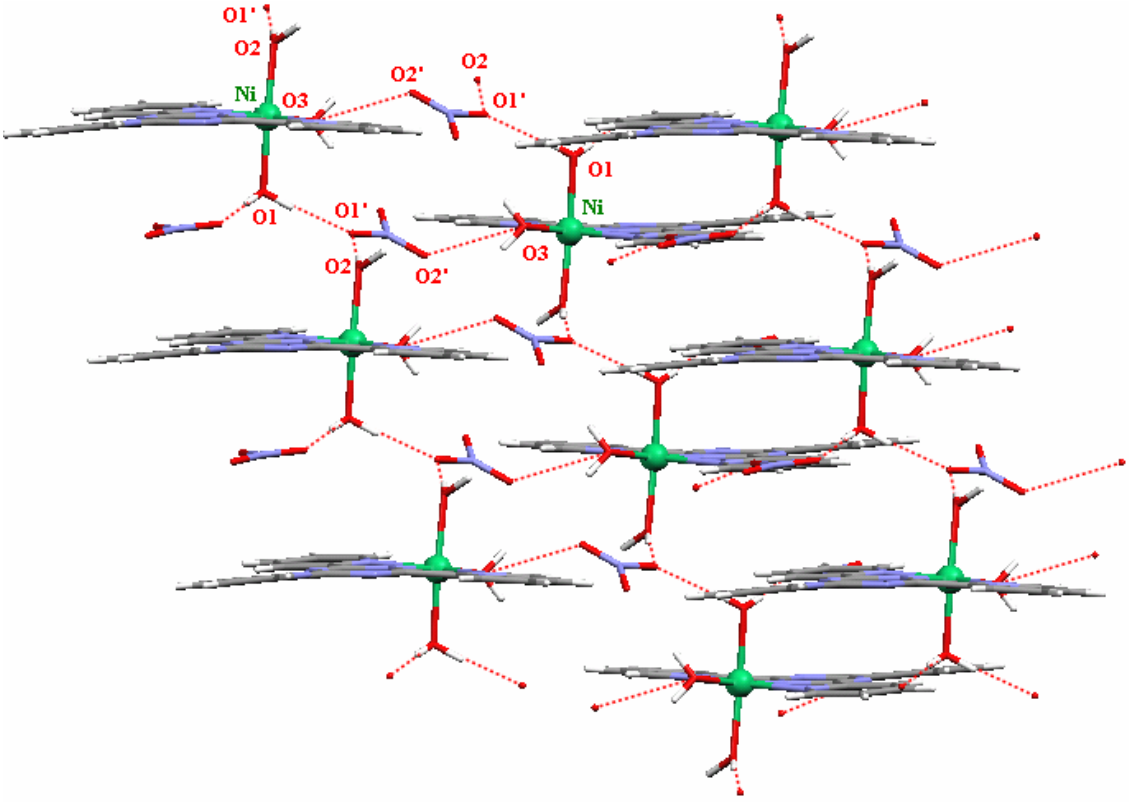


Figure 2.6. Hydrogen bonding network and π-π interactions within the lattice of Structure **D**.

Two different complexes (**2.8** and **2.9**) crystallised in the unit cell of Structure **B**. These complexes are shown later in Section 2.4 (Figure 2.8). The coordination sphere of the first complex, the cation **2.8**, consists of a tpt ligand, two water molecules, and one chloride ligand, whereas that of the second complex, **2.9**, contains a tpt ligand, **1.70**, one water molecule and two chloride ligands. Both complexes have a chloride ligand and a water ligand in the axial sites, and the remaining ligand, a water molecule (complex **2.8**) or a chloride ion (complex **2.9**) is coordinated *trans* to the triazine donor of ligand **1.70**. Again there is an extensive network of hydrogen bonds involving both lattice and coordinated water molecules and chloride ions, including one that links the axial coordinated water molecule of complex **2.8** to the axial chloride ligand of complex **2.9** (Cl1…O1′ 3.063(2) Å). Some of the hydrogen bonding interactions are shown by dotted lines later in Section 2.4 (Figure 2.8). Given the observed lability of the system, these hydrogen bonds may have some influence on the preference of chloride and water ligands for particular coordination sites in the isolated material. Some π-π interactions are also observed.

A view of complex **2.10** is shown later in Section 2.4 (Figure 2.10). This complex is like complex **2**.**7** in that it has two water ligands and one chloride ligand in addition to the ligand **1.70**. However, in this case the water ligands are *trans* to each other, and the chloride ligand is *trans* to the triazine ring (the same coordination environment is found in complex **2.11** in the incomplete Structure **I**). The observation of these different ligand configurations in the different crystal structures may well be tied to the ability of the complexes with different configurations to form hydrogen bonding networks within the constraints of the crystal structure packing. The other major difference is that the uncoordinated pyridine ring is protonated and closer to

being coplanar with the other aromatic rings. Presumably the planarity is a result of an interaction between the proton and the adjacent triazine ring nitrogen atom.

2.4. X-ray Crystallography

2.4.1. Crystal Structure of $[Ni(tpt)(H_2O)_3)]Cl_2.3H_2O$, 2.7, (Structure A)

Vapour diffusion of acetone into the aqueous solution of the complex resulted in the formation of large blocks of green crystalline material of **2.7** over a few days which were suitable for X-ray crystallography. A view of complex **2.7** (Structure **A**) with the atom-numbering scheme is shown in Figure 2.7. The geometry of the Ni(II) ion can be described as a distorted octahedron formed by the co-ordination of one tpt ligand, **1.70**, which functions as a tridentate ligand with nitrogen donor atoms in a meridional fasion. Three co-ordinated water molecules complete the octahedral environment of the dication. Three solvated water molecules and two Cl^- counter-ions stabilize the structure of the dication in the asymmetric unit. The distance between the Ni and the triazine nitrogen donor N1 is 1.977(4) Å; whereas the distances with the two pyridyl nitrogen donors N4 and N5 are long at 2.162(4) Å and 2.160(4) Å, respectively. The Ni-O distances vary from 2.051 to 2.073 Å; the longest Ni-O3 bond occurs when there is a strong H-bonding interaction with the lattice water molecules (O3-H3A…O5, O3-H3B…O4, and O3-H3B…O5 at 2.621(7), 3.031(6), and 3.213(8) Å, respectively) and the shortest Ni-O bond distance is that to the oxygen atom in the axial position (O1).

There is an extensive hydrogen bonding network involving the lattice water molecules and the chloride counterions.

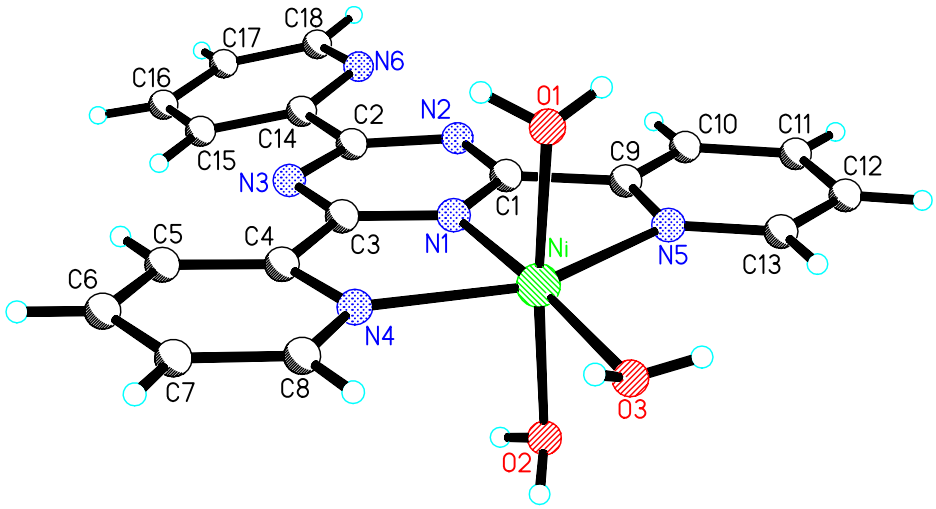


Figure 2.7. Molecular structure of the chloride salt of the triaqua complex **2.7** (Structure **A**) showing the numbering scheme adopted. (Structure **A**). Three solvated woter molecules and two chloride anions are omitted for clarity. Selected bond lengths [Å] and angles [°]: Ni-N(1) 1.977(4); Ni-O(2) 2.051(4); Ni-O(1) 2.063(4); Ni-O(3) 2.073(4); Ni-N(5) 2.160(4); Ni-N(4) 2.162(4); N(1)-Ni-O(2) 92.09(14); N(1)-Ni-O(1) 93.28(15); O(2)-Ni-O(1) 174.13(14); N(1)-Ni-O(3) 176.00(16); O(2)-Ni-O(3) 84.59(15); O(1)-Ni-O(3) 90.13(16); N(1)-Ni-N(5) 76.79(15); O(2)-Ni-N(5) 88.60(14); O(1)-Ni-N(5) 90.34(14); O(3)-Ni-N(5) 105.31(16); N(1)-Ni-N(4) 76.82(15); O(2)-Ni-N(4) 94.08(14); O(1)-Ni-N(4) 89.43(14); O(3)-Ni-N(4) 101.14(16); N(5)-Ni-N(4) 153.55(15).

2.4.2. Crystal Structure of [[Ni(tpt)Cl(H_2O)$_2$]Cl, 2.8, + [Ni(tpt)Cl$_2$(H_2O)], 2.9,].4H_2O (Structure B)

Orange needle-like crystals of **2.8** and **2.9**, which were suitable for X-ray crystallography, were obtained over a week from the same reaction mixture in which the crystalline materials of **2.7** were first formed. A perspective view of complexes **2.8** and **2.9** (Structure **B**) with the atom numbering is presented in Figure 2.8. The molecular geometries of two different Ni(II) complex ions which are cocrystallized in *P*$\bar{1}$ space group can be described as distorted octahedral. Ligand **1.70** acts as a tridentate ligand with nitrogen donor atoms in a meridional fashion. Three nitrogen atoms from the ligand (N1, N4, and N5) and one chloride ion (Cl2) form the equatorial plane, and one oxygen atom from a coordinated water molecule (O1) along with one chloride ion (Cl1) are in axial positions. Cl2 is stabilized in the equatorial position through a strong hydrogen bond to a solvated water molecule (O11-H11A…Cl2) (Figure 2.8). Cl1 is also hydrogen bonded to a coordinated water molecule in the other dication (O1′-H1B′…Cl1). In the second dication, three nitrogen atoms from the ligand (N1′,N4′, and N5′) and one oxygen atom from a coordinated water molecule (O2′) are in the equatorial plane. One oxygen atom from a second coordinated water molecule (O1′) and a chloride ion (Cl1′) which are in axial position completing the distorted octahedral environment of the Ni′(II) ion. The coordinated water molecules are both hydrogen bonded to a chloride counterion within the lattice (O1′-H1A′…Cl10 and O2′-H2B′…Cl10).

The distance between the Ni and the triazine nitrogen atom (N1) is 1.989(2) Å; whereas the distances with the two pyridyl nitrogen donors N4 and N5 are long at

2.144(2) Å and 2.145(2) Å, respectively; the longest distance of Ni-Cl1 could be due to strong H-bonding interaction with the water molecule (O1′).

The bond distance of Ni′ to the middle nitrogen (N1′) (1.987(2) Å) is significantly shorter (by about 0.17 Å) than Ni′-N4′ (2.171(2) Å) and Ni′-N5′(2.151(2) Å) distances. Ni′-Cl1′ distance (2.380(8) Å) is shorter than the Ni-Cl1 (2.470(8)Å) again presumably due to H-bonding interaction of Cl1 with the water molecule (O1′). The Ni′-O′ distances vary from 2.054(18) Å to 2.086(2) Å; the longest distance of Ni′-O1′ could be due to strong H-bonding interaction with the chloride (Cl1). In the ligands **1.70**, the $C(sp^2)$-$C(sp^2)$ and $C'(sp^2)$-$C'(sp^2)$ distances within the ring are normal (average 1.403(4) Å) and the exterior bonds C1-C9, C3-C4, C2-C14 in the first dication and C1′-C9′, C3′-C4′, C2′-C14′ within the 2nd dication average to 1.486(3) Å and 1.485(3) Å, respectively.

The two dications are held together by hydrogen bonds with the equatorial chloride (Cl1) and the coordinated water molecule (O1′) (Cl1…O1′ 3.063(2) Å). The Ni-Ni′ distance is 6.58 Å. As it is shown in Figure 2.8 in contrast to complex **2.7**, again there is an extensive hydrogen bonding network involving the lattice water molecules and the chloride counterions. The packing is also governed by weak π-π interactions.

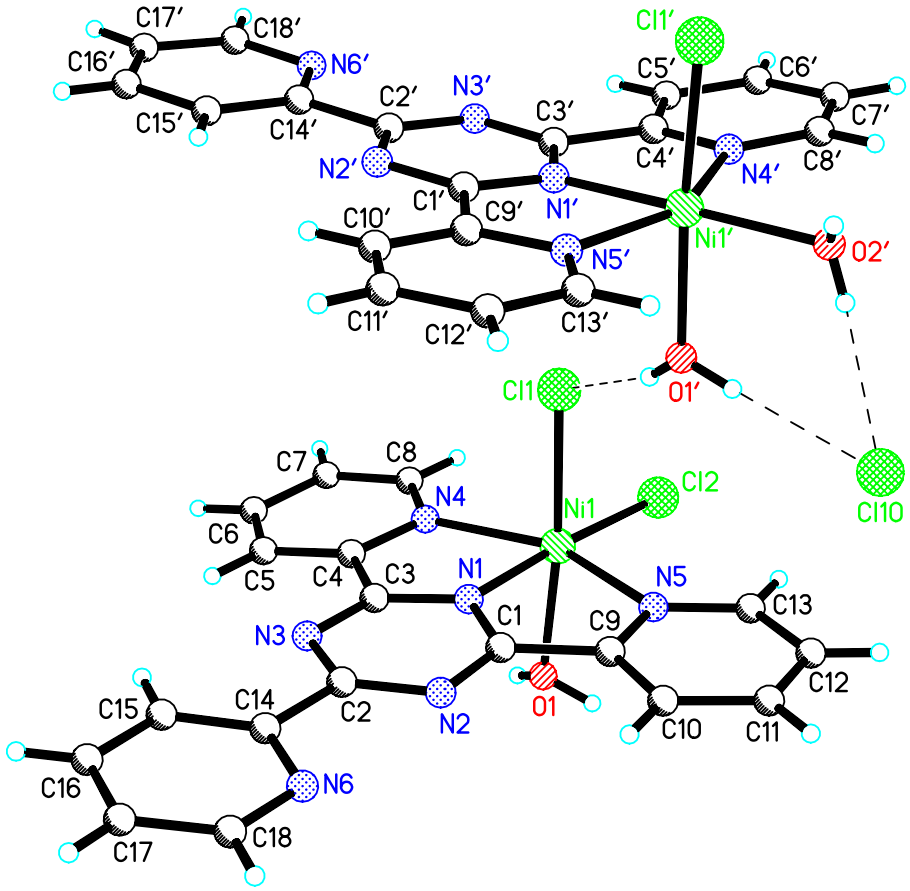


Figure 2.8. Structure **B**, comprised of complex **2.8** (top) and complex **2.9** (bottom). The dotted lines indicate hydrogen bonding in the lattice. Selected bond lengths [Å] and angles [°]: Ni(1)-N(1) 1.989(2); Ni(1)-O(1) 2.1148(19); Ni(1)-N(4) 2.144(2); Ni(1)-N(5) 2.145(2); Ni(1)-Cl(2) 2.3113(8); Ni(1)-Cl(1) 2.4695(8); Ni(1')-N(1') 1.987(2); Ni(1')-O(2') 2.0535(18); Ni(1')-O(1') 2.086(2); Ni(1')-N(5') 2.151(2); Ni(1')-N(4') 2.171(2); Ni(1')-Cl(1') 2.3801(8); N(1)-Ni(1)-O(1) 84.64(8); O(1)-Ni(1)-N(4) 84.49(8); O(1)-Ni(1)-N(5) 90.54(8); O(1)-Ni(1)-Cl(2) 92.35(6); N(4)-Ni(1)-Cl(2) 104.76(6); N(5)-Ni(1)-Cl(2) 101.55(6); N(1)-Ni(1)-Cl(1) 90.64(6); O(1)-Ni(1)-Cl(1) 174.35(6); N(4)-Ni(1)-Cl(1) 91.42(6); N(5)-Ni(1)-Cl(1) 91.39(6); Cl(2)-Ni(1)-Cl(1) 92.47(3); N(1')-Ni(1')-O(2') 176.06(8);

N(1')-Ni(1')-O(1') 91.53(8); O(2')-Ni(1')-O(1') 86.42(8); O(2')-Ni(1')-N(5') 106.29(8); O(1')-Ni(1')-N(5') 89.73(9); O(2')-Ni(1')-N(4') 100.58(8); O(1')-Ni(1')-N(4') 92.82(9); N(1')-Ni(1')-Cl(1') 96.01(6); O(2')-Ni(1')-Cl(1') 86.37(6); O(1')-Ni(1')-Cl(1') 170.55(6); N(5')-Ni(1')-Cl(1') 86.43(6); N(4')-Ni(1')-Cl(1') 94.51(6).

2.4.3. Crystal Structure of $[Ni(Htpt)Cl(H_2O)_2]Cl_2.2H_2O$, 2.10, (Structure C)

Orange needle-like crystals of **2.10** suitable for X-ray diffraction were obtained by vapour diffusion of acetone into the aqueous solution of the complex over a week. Single crystals of **2.10** with different morphology also were obtained by slow evaporation of acetone from the solution containing the needle-like crystalline materials (see Figure 2.9). A view of complex **2.10** with the atom numbering scheme is shown in Figure 2.10. The asymmetric unit consists of a cation, two chloride counterions and two solvated water molecules. The Ni atom is in the centre of a distorted octahedron of one central triazine nitrogen atom and two pyridyl nitrogen atoms of the ligand (N1, N4, and N5), two axially coordinated water molecules, and one equatorially coordinated chloride ion.

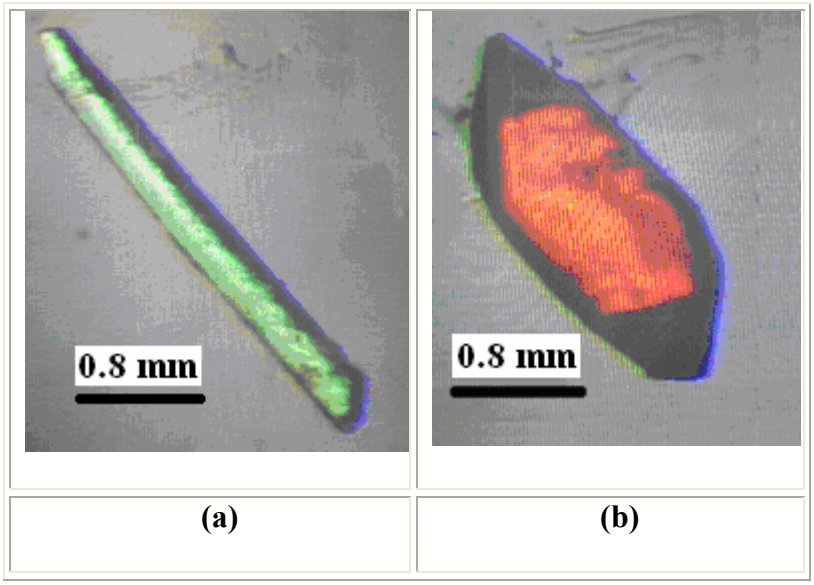


Figure 2.9. Two different crystal morphologies of complex **2.10** in Structure **C. (a)** Orange needle-like single crystal of structure **C** obtained by vapour diffusion of acetone into the aqueous solution of the complex over a week (the green colour observed on the microscope monitor is due to the transmission of polarised light). **(b)** Orange prism single crystal of the structure **C.**

It is interesting to note that all the aromatic rings in the ligand are coplanar. The coplanarity of the central triazine ring and the two pyridyl ring coordinated to the Ni ion through the nitrogen atoms is obviously due to coordination to the Ni atom in a meridional fashion. The coplanarity of the terminal pyridyl ring which is also protonated could be due to the interaction of the proton on the protonated nitrogen (N6) with the nitrogen of the central ring (N3). This new hydrogen bonding interaction can overcome the existing interaction of the hydrogen on the carbon (C15) with the other central triazine nitrogen atom (N). Furthermore, the hydrogen bonding interaction of the chloride counter-ion (Cl3) with the proton on the protonated nitrogen

(N6) favours the coplanarity of the end protonated ring. The Cl3-Ni6 distance is the shortest Cl-N interaction distance (N6-H6X…Cl3 at 3.144(19) Å within the lattice.

The distance between the Ni atom and the triazine nitrogen donor N1 is 2.013(16) Å; whereas the distances with the two pyridyl nitrogen donors N4 and N5 are long at 2.157(17) Å and 2.178(17) Å, respectively. The chloride ion (Cl1) coordinated to Ni(II) ion favours the equatorial position around the Ni(II) ion, presumably due to a strong hydrogen bonding network within the cell. In contrast to structures **A** and **B**, the dications are held together by the strong hydrogen bondings shared between the axially coordinated water molecules, chloride counter-ions, and the coordinated chloride ions along with the hydration water molecules dispersed throughout the crystal lattice. There are also weak π-π interactions within the lattice.

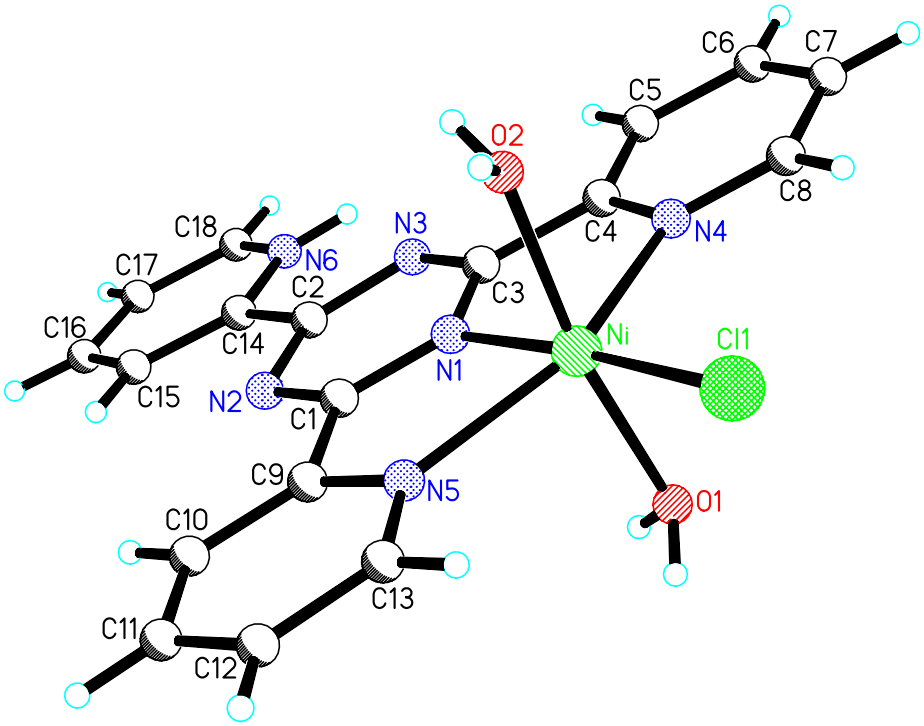


Figure 2.10. Crystal and molecular structure of complex **2.10** (Structure **C**) showing the numbering scheme adopted. Two water molecules and two chloride

anions are omitted for clarity. Selected bond lengths [Å] and angles [°]: Ni-N(1) 2.0126(16); Ni-O(2) 2.0768(16); Ni-O(1) 2.0837(15); Ni-N(4) 2.1566(17); Ni-N(5) 2.1778(17); Ni-Cl(1) 2.3206(6); N(1)-Ni-O(2) 92.45(6); N(1)-Ni-O(1) 91.14(6); O(2)-Ni-O(1) 171.43(7); N(1)-Ni-N(4) 76.57(6); O(2)-Ni-N(4) 84.85(7); O(1)-Ni-N(4) 88.43(6); N(1)-Ni-N(5) 76.03(6); O(2)-Ni-N(5) 95.16(7); O(1)-Ni-N(5) 93.25(6); N(4)-Ni-N(5) 152.57(6); N(1)-Ni-Cl(1) 175.90(5); O(2)-Ni-Cl(1) 89.42(5); O(1)-Ni-Cl(1) 87.50(4); N(4)-Ni-Cl(1) 107.25(5); N(5)-Ni-Cl(1) 100.17(5).

2.4.4. Crystal Structure of $[Ni(tpt)(H_2O)_3](NO_3)_2$, 2.12, (Structure D)

A perspective view of complex **2.12** with the atom numbering scheme is presented in Figure 2.11. The geometry of the Ni(II) ion can be again described as a distorted octahedron formed by the co-ordination of one tpt ligand, **1.70**, which functions as a tridentate ligand with nitrogen donor atoms in a meridional fashion. Three co-ordinated water molecules complete the octahedral environment of the dication. Two NO_3^- counter-ions stabilize the structure of the dication in the asymmetric unit. The distance between the Ni and the triazine nitrogen donor N1 is 1.993(2) Å; whereas the distances with the two pyridyl nitrogen donors, N4 and N5, are long at 2.148(2); 2.162(2) Å, respectively. The Ni-O distances vary from 2.045(2) to 2.101(2)Å; the longest distance of Ni-O1 bond could be due to strong H-bonding interaction with the NO_3^- anions (O1-H1A…O2", O1-H1A…O2", O1-H1A…N", and O1-H1B...O1' at 2.807(3), 3.304(4), 3.468(4), and 2.786(3) Å, respectively) and the shortest Ni-O bond distance is that to the oxygen atom in the *trans* position (O3).

There is an extensive hydrogen bonding network involving the nitrate counterions. There are also π-π stacking interactions between the dications in the structure. The separation between the planes of the central triazine rings of the adjacent molecules (face-face) is 3.6 Å (centroid-centroid).

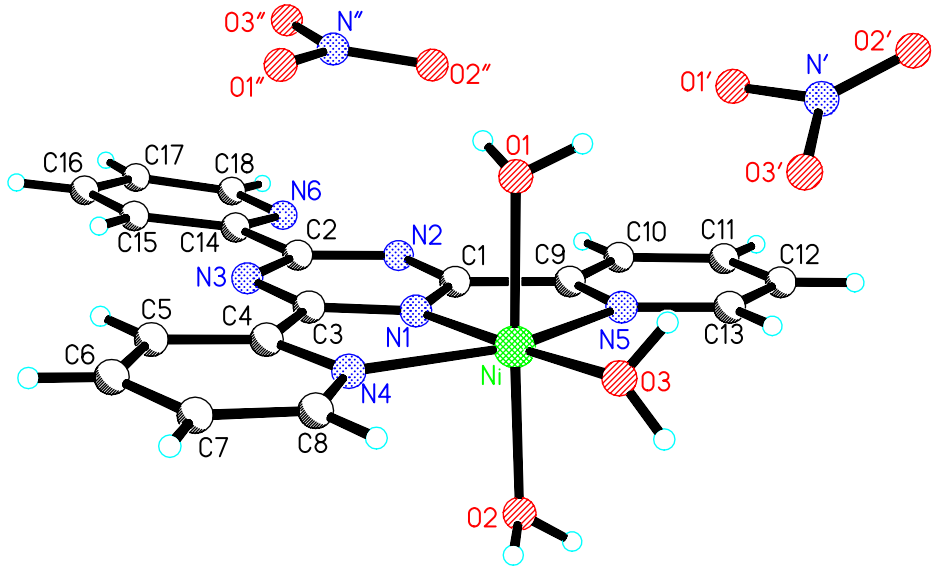


Figure 2.11. Crystal structure of complex **2.12** (Structure **D**). Selected bond lengths [Å] and angles [°]:Ni-N(1) 1.993(2); Ni-O(3) 2.045(2); Ni-O(2) 2.090(2); Ni-O(1) 2.101(2); Ni-N(4) 2.148(2); Ni-N(5) 2.162(2); N(1)-Ni-O(3) 175.40(9); N(1)-Ni-O(2) 92.96(9); O(3)-Ni-O(2) 90.64(9); N(1)-Ni-O(1) 91.08(9); O(3)-Ni-O(1) 85.29(9); O(2)-Ni-O(1) 175.90(8); N(1)-Ni-N(4) 77.22(9); O(3)-Ni-N(4) 99.93(10); O(2)-Ni-N(4) 89.88(9); O(1)-Ni-N(4) 90.33(9); N(1)-Ni-N(5) 76.90(9); O(3)-Ni-N(5) 105.91(10); O(2)-Ni-N(5) 91.27(9); O(1)-Ni-N(5) 90.32(9); N(4)-Ni-N(5) 154.12(10).

2.4.5. Crystal Structure of $[Ni(tpt)_2](ClO_4)_2$, 2.13, (Structure E)

Crystals of **2.13,** which were suitable for X-ray diffraction, were obtained by vapour diffusion of MeOH into DMSO solution of the complex. The molecular structure of complex **2.13** (Structure **E**) with numbering scheme is presented in Figure 2.12. The nickel is in the expected distorted octahedral geometry, with each of the tridentate ligands **1.70** occupying three meridionally arranged coordination sites.

The Ni-N bonds to the central triazine rings are significantly longer than those were observed for complexes **2.7, 2.8, 2.9** and **2.10** as well as for the literature complex $[Ni(terpy)_2](PF_6)_2$ (2.016 and 2.009Å).[187,356] The Ni-N bonds to the side pyridine rings (2.211, 2.216, 2.220, and 2.225Å) are also longer than those of complexes **2.7, 2.8, 2.9** and **2.10,** as well as those of complex $[Ni(terpy)_2](PF_6)_2$[187,356] (2.136, 2.125, 2.107, 2.114, and 2.121Å, respectively).

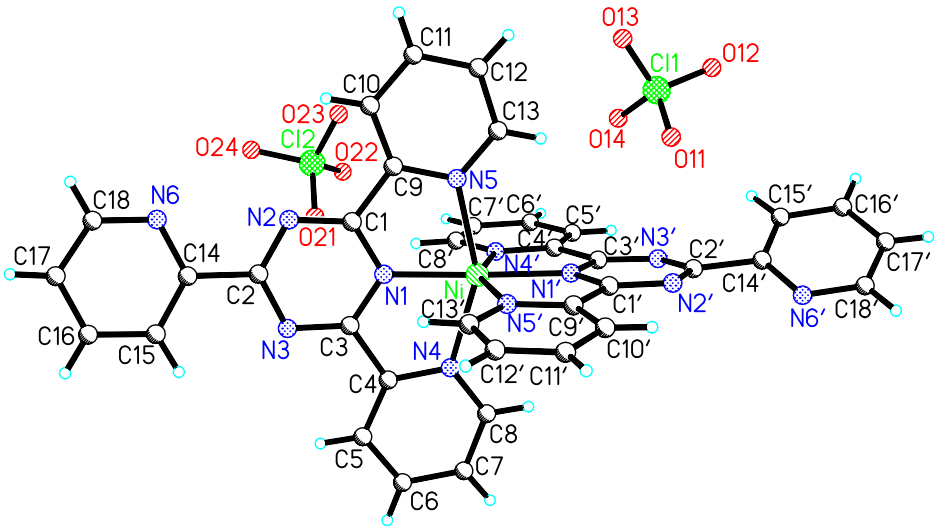


Figure 2.12. Molecular structure of complex **2.13** (Structure **E**) with numbering scheme depicted. Selected bond lengths [Å] and angles [°]:Ni-N(1) 2.053(3); Ni-

N(1') 2.054(2); Ni-N(4') 2.211(3); Ni-N(5') 2.216(3); Ni-N(5) 2.220(3); Ni-N(4) 2.225(3); N(1)-Ni-N(1') 177.21(10); N(1)-Ni-N(4') 105.41(10);N(1')-Ni-N(4') 76.35(9); N(1)-Ni-N(5') 101.70(10); N(1')-Ni-N(5') 76.62(10); N(4')-Ni-N(5') 152.84(9); N(1)-Ni-N(5) 76.67(10); N(1')-Ni-N(5) 101.14(10); N(4')-Ni-N(5) 93.68(10); N(5')-Ni-N(5) 93.96(9); N(1)-Ni-N(4) 76.03(10); N(1')-Ni-N(4) 106.20(10); N(4')-Ni-N(4) 91.95(9); N(5')-Ni-N(4) 93.11(10); N(5)-Ni-N(4) 152.65(10).

2.4.6. Crystal Structure of $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, 2.15, (Structure F)

Green needles suitable for X-ray diffraction crystallography were obtained from the reaction mixture. A perspective view of complex **2.15** with the atom numbering is presented in Figure 2.13. Two Ni(II) ions are bridged by ligand **1.70**. Ni1 is coordinated to ligand tpt, **1.70**, in a tridentate terpyridine-like fashion with a distorted octahedral geometry; whereas Ni2 is coordinated to **1.70** in a bidentate bipyridine-like moiety. The triazine nitrogen atom N1 along with the side pyridine nitrogen atoms (N4 and N5) and the oxygen atom O2 of the ethanol molecule bound to Ni1 are located in the equatorial plane. The axial bonds of Ni1 to the other oxygen atoms (O1 from a water molecule and O71 of a nitrate anion) complete the distorted octahedral geometry around the Ni1(II) ion. On the other side, the equatorial plane of the Ni2 environment is defined by a nitrogen atom of the terminal pyridine ring (N6), two oxygen atoms O91 and O92 provided by a bridged nitrate anion (N9) and one oxygen atom O81 of

the other coordinated nitrate anion (N8). The triazine ring nitrogen atom N2 bond to the Ni2 alond with the bond formed between an oxygen atom O3 of the second coordinated ethanol molecule in the complex are situated in the axial position to the Ni2(II) ion environment. The equatorial plane of Ni1 is coplanar to the central triazine ring while the equatorial plane of Ni2 is nearly perpendicular to the central ring. (O81-Ni2-N2 (94.68°) and N2-Ni2-O91 (89.49°)).

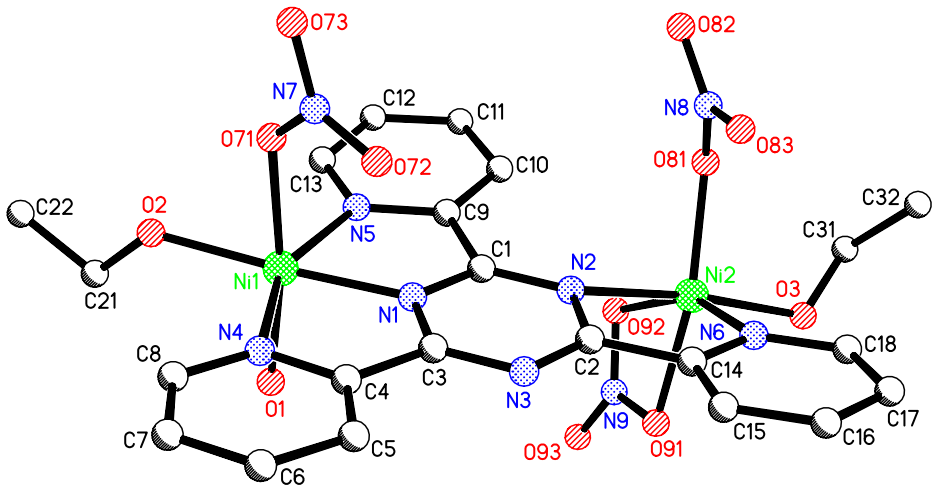


Figure 2.13. Molecular structure of the dinickel complex **2.15** cation (Structure **F**). The hydrogen atoms and a nitrate anion are omitted for clarity. Selected bond lengths [Å] and angles [°]: Ni(1)-N(1) 1.969(3); Ni(1)-O(2) 2.007(3); Ni(1)-O(1) 2.051(3);Ni(1)-O(71) 2.078(2); Ni(1)-N(5) 2.110(3); Ni(1)-N(4) 2.118(3); Ni(2)-O(81) 2.007(2); Ni(2)-N(6) 2.012(3); Ni(2)-O(3) 2.047(3); Ni(2)-O(91) 2.076(2); Ni(2)-O(92) 2.102(2); Ni(2)-N(2) 2.179(3); N(1)-Ni(1)-O(2) 176.95(12); N(1)-Ni(1)-O(1) 92.57(12); O(2)-Ni(1)-O(1) 89.67(12); N(1)-Ni(1)-O(71) 96.60(10); O(2)-Ni(1)-O(71) 81.05(11); O(1)-Ni(1)-O(71) 170.22(11); N(1)-Ni(1)-N(5) 76.56(12); O(2)-Ni(1)-N(5) 101.42(12); O(1)-Ni(1)-N(5) 88.74(11); O(71)-Ni(1)-N(5) 90.07(10); N(1)-Ni(1)-N(4) 78.60(12); O(2)-Ni(1)-N(4) 103.40(12); O(1)-

Ni(1)-N(4) 92.35(11); O(71)-Ni(1)-N(4) 92.80(10); N(5)-Ni(1)-N(4) 155.16(12); O(81)-Ni(2)-N(6) 106.25(10); O(81)-Ni(2)-O(3) 91.76(10); N(6)-Ni(2)-O(3) 93.62(12); O(81)-Ni(2)-O(91) 158.87(10); N(6)-Ni(2)-O(91) 94.87(10); O(3)-Ni(2)-O(91) 85.99(10); O(81)-Ni(2)-O(92) 97.18(10); N(6)-Ni(2)-O(92) 156.15(11); O(3)-Ni(2)-O(92) 90.01(10); O(91)-Ni(2)-O(92) 61.85(10); O(81)-Ni(2)-N(2) 94.68(11); N(6)-Ni(2)-N(2) 80.53(12); O(3)-Ni(2)-N(2) 172.29(10); O(91)-Ni(2)-N(2) 89.49(10); O(92)-Ni(2)-N(2) 93.37(10).

As it is shown in the Figure 2.14, the five-membered-ring formed by the coordination of Ni2 to the nitrogen atoms in tpt ligand, **1.70**, (Ni2-N2-C2-C14-N6) is not coplanar with the central triazine. The angle between the five-membered-ring and the side pyridine ring (N5-C9-C10-C11-C12-C13) is 20.4°. Ni1-Ni2 bond distance is 6.171Å.

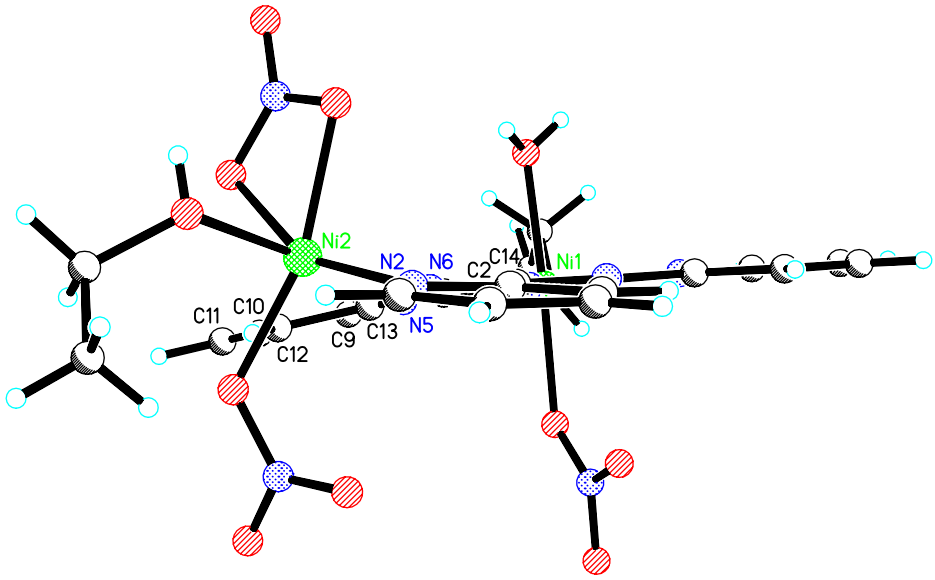


Figure 2.14. A view of Complex **2.15** cation (Structure **F**) which represents the angle between Ni2-N2-C2-C14-N6 five-membered-ring plane and the side pyridine ring N5-C9-C10-C11-C12-C13 to be 20.4°.

2.5. Conclusion

A series of nickel(II) complexes of 2,4,6-tris(2-pyridyl)-1,3,5-triazine, **1.70**, has been prepared and identified using crystallographic techniques. Changes in the reaction conditions result in complexes with metal-triazine ligand ratios of 2:1, 1:1, and 1:2 being isolated. The results of the structural studies show that different stereoisomers can be formed, and the structures have been examined in order to identify the stabilising features which might have led to these particular compounds being formed in preference to others in such labile systems. Numerous π-π stacking and hydrogen bonding interactions can be found within the lattices of the structures,

but these are notably absent in the structure of $[Ni(tpt)_2](ClO_4)_2$, **2.13**, the complex isolated when perchlorate ions are added to solutions of all the other compounds.

The tpt ligand, **1.70**, can form a range of complexes with nickel(II), depending on the other ligands, anions and solvents that are present in the reaction mixture. While the formation of particular complexes or mixtures of complexes can be understood based on general principles of reactivity in labile systems, it is not possible to predict just which complex(es) will be isolated under a given set of conditions. Many of the crystal structures appear to be stabilised by π-π stacking and hydrogen bonding interactions, and presumably this affects which complex (and which stereoisomer) is formed under any given set of conditions.

This Chapter has demonstrated that the ligand tpt is a potential spacer, which can function simultaneously as a tridentate and a bidentate ligand. Some dinuclear Ni(II) complexes of tpt ligand including $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15**, were prepared and crystallographically characterised. To the best of our knowledge, there is no other report of crystallographic characterisation of a dinuclear Ni(II) complex containing tpt ligand.

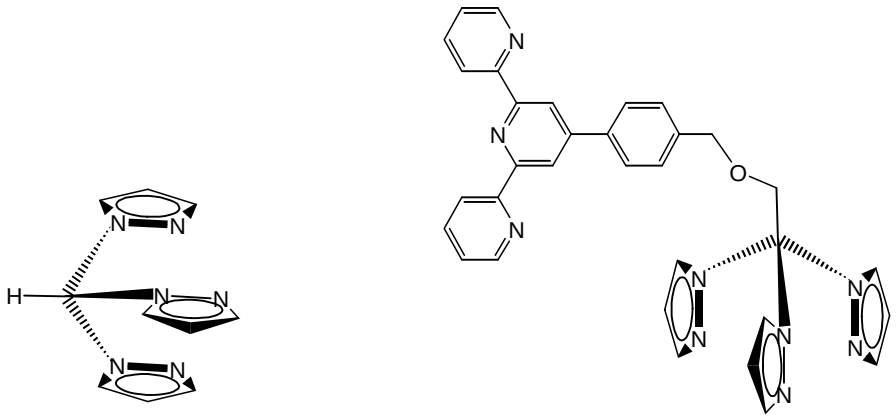
Attempts to synthesise target dinuclear Ru(II)-Co(III) complexe **2.3** proved unsuccessful under our conditions. One of the possible reasons that makes mononuclear Ru(II) complexes containing tpt ligand unfavourable towards Co(III) ions, could be the steric hindrance around the bidentate site of the ligand. Later in this thesis, however, we will introduce new bridging ligands based on terpyridyl systems with two remote binding sites. This approach may facilitate binding Co(III) ion at one of the sites of each ligand, while the other binding domain (terpyridyl site) bound to Ru(II) ion.

Chapter 3

Synthesis and Structural Characterization of Some Polypyridyl and Tris(1H-pyrazol-1-yl)methane Ruthenium(II) Complexes

3.1. Introduction

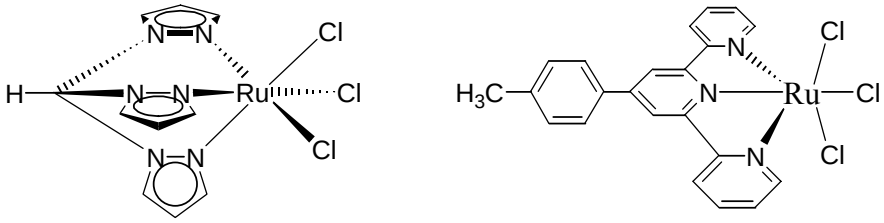
The initial motivation for the work reported in this chapter was to establish the conditions under which a tris(1*H*-pyrazol-1-yl)methane (tpm) derivative could be induced to coordinate to a ruthenium centre that already had a (tpm) ligand attached to it. The intention was to conduct a model study to guide my work with a more complicated and less readily available ditopic terpyridine-tris(1*H*-pyrazol-1-yl)methane ligand, 4'-(4-(2,2,2-tris(1*H*-pyrazol-1-yl)ethoxymethyl)phenyl)-2,2':6',2"-terpyridine (pzt)[348], **1.71** (Figure 3.1).



tpm, **3.1** pzt, **1.71**

Figure 3.1. Ligands tpm and pzt[348].

In particular, we hoped that we would be able to achieve regioselective coordination to the ditopic ligand through the use of metal complexes that already had either facial tridentate or meridional tridentate ligands attached to the metal centre. If a facial tridentate ligand is attached to a metal centre, the remaining three coordination sites on an octahedral metal centre must also be disposed in a facial manner. In principle, therefore, the known compound [Ru(tpm)Cl_3], **3.2**,[194,385] (Figure 3.2) should be restricted to the tpm binding site of the ditopic ligand, if it is to replace all three chloride ligands with heterocyclic donors. On the other hand, a 4'-(4-toluyl)-2,2':6',2''-terpyridine (ttp) complex, *e.g.* [Ru(ttp)Cl_3], **3.3**,[386] (Figure 3.2) might be expected to bind to the terpyridyl binding site. In these kinds of reaction, reduction of the ruthenium centre usually occurs during the ligand exchange reaction. The chemistry of the terpyridine type systems is well established,[121,122,143,146,156,183,187,189,193,195,199,203,206,212,213,215,219,221,222,224-227,230,233,244,356,358,360-362,382,383,387-395] but much less work has been done on tpm based systems.[263,264,266,268,270,272,275,278,282,396]



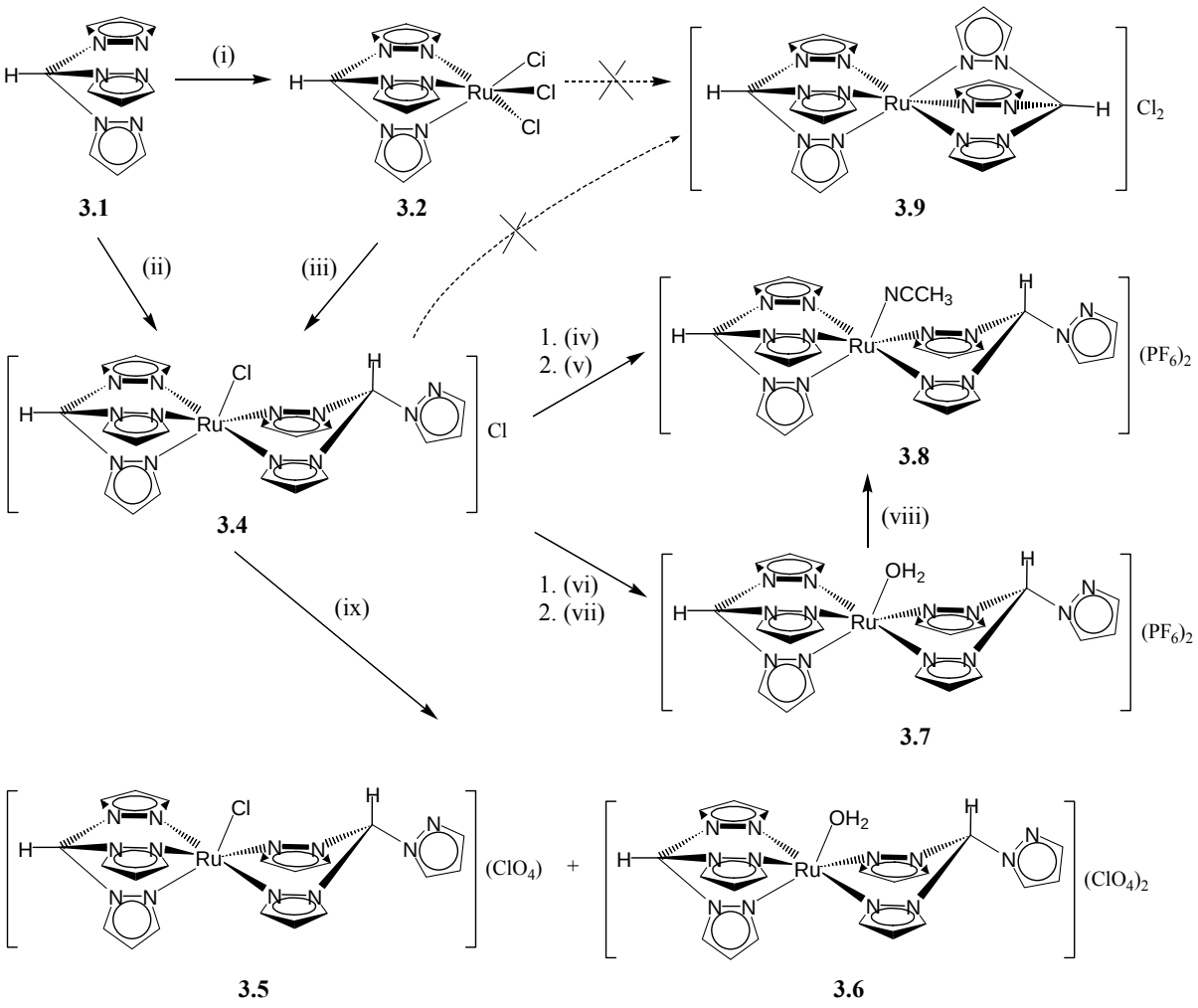
3.2 **3.3**

Figure 3.2. Ru(III) complexes of tpm and ttp ligands, **3.2** and **3.3**, respectively.

This chapter describes the synthesis and structural characterisation of some Ru-tpm complexes, and also the results of some structural studies on closely related ttp systems.

3.2. Results and Discussion

The coordination chemistry of the ruthenium(II)-tpm system that we have explored is shown in Scheme 3.1. The reaction of Ru(tpm)Cl_3, **3.2**, with tpm ligand, **3.1**, afforded a green powder which was collected from a dark blue reaction mixture. ^{1}H NMR studies on solutions of the green powder sample showed immediately that the green complex was not the bis-tpm complex, **3.9**, that might have been expected if all six pyrazolyl groups of two tpm ligands were coordinated to the ruthenium centre.



Scheme 3.1. (i) $RuCl_3.3H_2O$, EtOH, reflux, 4h; (ii) $RuCl_3.3H_2O$ or $[Ru(ph\text{-}CN)_4Cl_2]$ (2: 1), EtOH, reflux, 15 min.; (iii) tpm (1 equimolar), EtOH: water (3: 1), reflux, 10 min; (iv) $AgClO_4$, dry CH_3CN, under Ar, reflux, 2 h; (v) NH_4PF_6; (vi) $AgClO_4$, acetone: water (3: 1), reflux, 2 h; (vii) NH_4PF_6; (viii) CH_3CN, acetone: water (3: 1), reflux, 24 h; (ix) $AgClO_4$, acetone, under Ar, reflux, 2 h.

The ^1H NMR spectrum, shown in Figure 3.3, contained 14 resonances: twelve in the aromatic region that can be assigned to four sets of pyrazolyl ring protons, and two singlets at around 10.3 and 10.6 ppm assigned to the CH groups of two tpm ligands. Two of the sets of pyrazolyl ring proton signals had integrations twice the size of the other two sets. The ^{13}C NMR data were entirely consistent with these results.

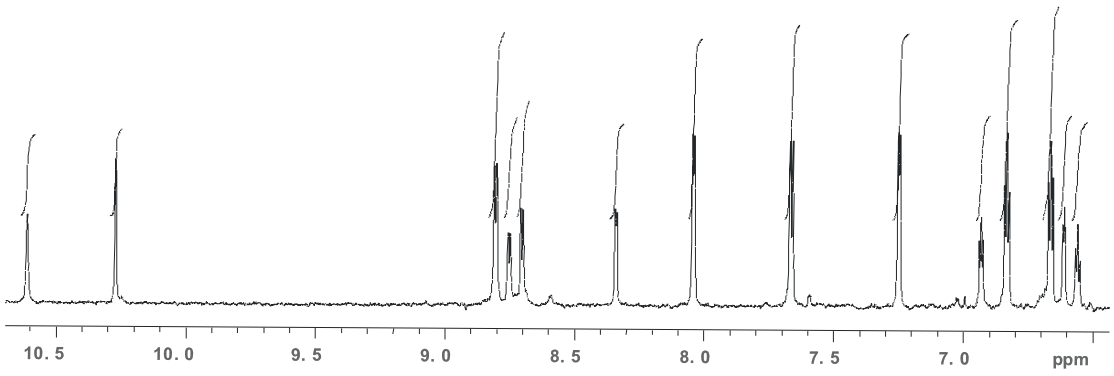


Figure 3.3. ^{1}H NMR spectra of [Ru(tpm)$_2$Cl] Cl, **3.4**, in dmso-d_6 solution.

At least two tpm ligands are clearly coordinated to the metal centre, based on the number of NMR signals that are observed, and an ESI-MS isotope pattern for a singly charged ion at around 565 units is consistent with a formulation of $[Ru(tpm)_2Cl]^+$ for the complex ion. The symmetry of the complex that is implied by the NMR data would result if one of the pyrazolyl groups of the second tpm ligand remained uncoordinated and the sixth coordination site was occupied by a chloride ligand. The isotope pattern for a 2+ species, $[Ru(tpm)_2Cl]^{2+}$, at around 265 units is consistent with that complex having lost a chloride ligand in the spectrometer.

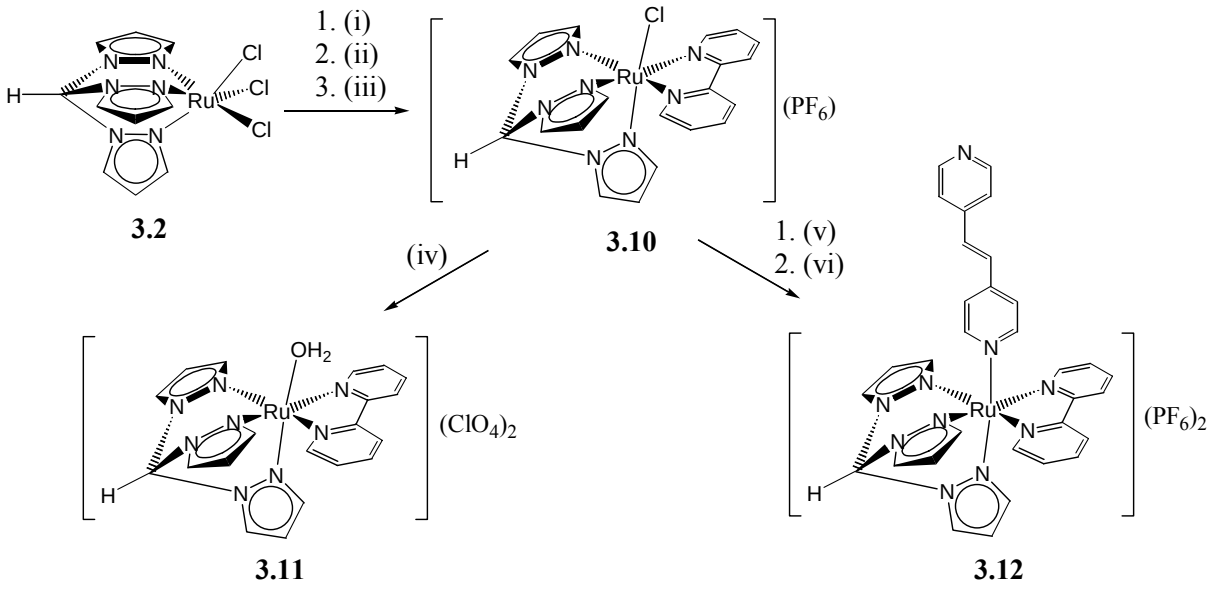
Overall, these data are consistent with the product being either structure **3.4** or the isomer where the methine proton of the bidentate tpm ligand is *anti* to the chloride ligand. We believe that **3.4** is the more likely structure for the complex, based on the results of a poorly refined X-ray crystal structure of compound **3.5** that was obtained during the synthesis of compounds **3.5** and **3.6**, and the large change in the chemical shift of one methine proton that was observed on exchanging the chloride ligand for a water ligand. In addition, if the complex were the other isomer there would seem to be no reason why the third pyrazolyl group of the second tpm ligand should not coordinate during the ligand exchange chemistry described below. The same complex,

3.4, was isolated when either $RuCl_3.3H_2O$ or $[Ru(phCN)_4Cl_2]$ were treated with two equivalents of the tpm ligand (Scheme 3.1).

Reaction of the green complex, **3.4,** with $AgClO_4.H_2O$ in aqueous acetone yielded a blue product which was isolated as the PF_6^- salt, by the addition of excess NH_4PF_6, after AgCl was filtered off. Only the starting chloro complex is recovered if the reaction is conducted in dry acetone. ^{1}H NMR spectra of the blue complex, **3.7**, contain 15 resonances: twelve resonances in the aromatic region for the four sets of pyrazolyl rings protons, two singlets at around 8.9 and 10 ppm for the CH methine group's hydrogens, and a two proton peak at 6.5 ppm that can be assigned to a coordinated water molecule. ESI-MS studies of the blue powder in CH_3CN solution show isotope patterns at *m/z* 693 and 274, that can be assigned to $\{[Ru(tpm)_2(OH_2)]PF_6\}^+$ and $[Ru(tpm)_2(OH_2)]^{2+}$, respectively, and these results are entirely consistent with removal of the chloride ligand and its replacement with a water ligand during the reaction. Similar chemistry can be conducted in acetonitrile solution and, under these conditions, the sixth coordination site is occupied by acetonitrile. The ^{1}H NMR spectrum of the complex in dmso-d_6, ESI-MS in CH_3CN, and IR of the solid material are all consistent with the presence of the acetontrile ligand and an uncoordinated pyrazole group.

These results clearly show that while it is possible to remove the chloride ligand, this only occurs if there is a suitable ligand to replace it. The pendant pyrazolyl group does not coordinate. In principle, this ligand substitution reaction could occur for both possible isomers of the isolated bis(tpm) complex, but the reaction will be more difficult for the isomer shown, **3.4**, because coordination of the pendant group can only occur if there is a rearrangement reaction within the coordination sphere.

Complexes [Ru(tpm)(bpy)Cl](PF_6), **3.10**, [Ru(tpm)(bpy)(OH_2)]$(ClO_4)_2$, **3.11**, and [Ru(tpm)(bpy)(bpe)]$(PF_6)_2$, **3.12**, (Scheme 3.2) provide a good basis with which to compare the properties of the series of bis(tpm) complexes, and were synthesized using literature procedures.[194,397] All complexes were characterized by NMR and ESI-MS techniques.



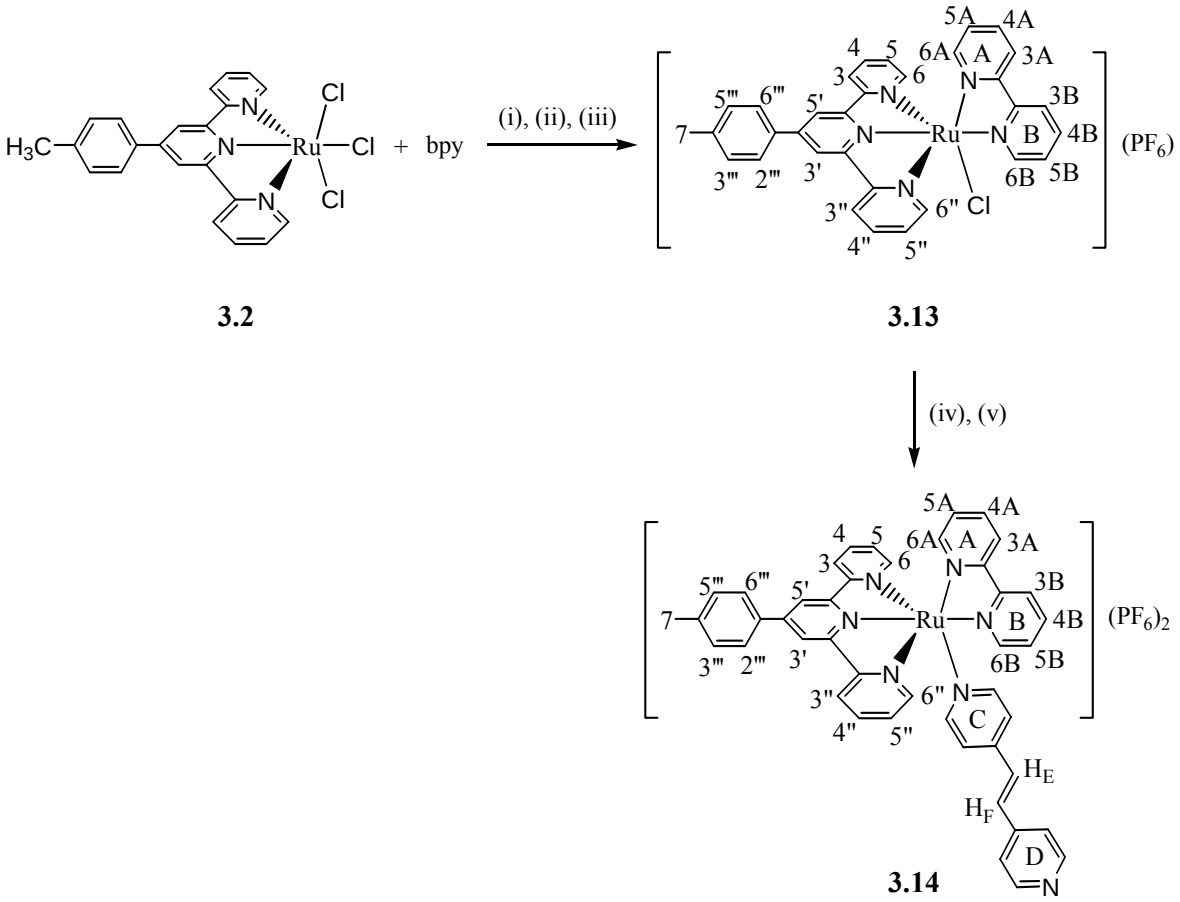
Scheme 3.2. (i) bpy, EtOH: water (3: 1), reflux, 5 min; (ii) LiCl, NEt_3, reflux, 10 min; (iii) NH_4PF_6; (iv) $AgClO_4$, acetone: water (3: 1), reflux, 2 h; (v) bpe, EtOH: water (1:1), reflux, under Ar, 6 h; (vi) NH_4PF_6.

The NMR data for the aqua complex **3.11** are similar to the reported values for this complex, the chloro complex **3.10** data are what might be expected, and ESI-MS data were also consistent with the proposed structures. The very small change in the NMR data on replacement of the chloride ligand with water in this pair of complexes shows that the nature of the monodentate ligand has very little effect on the chemical shift of the methine proton of a tridentate, facially coordinated tpm ligand. This

provides supporting evidence for the isomer assignment made for the bis(tpm) complex above. The large chemical shift change that is observed for one methine proton on exchanging a water ligand for a chloride ligand led us to assign those signals to the bidentate tpm ligand. Further, we conclude that the methine proton must have been in close proximity to the monodentate ligand for such large changes to be observed.

I was more fortunate with this series of Ru complexes in that crystalline material was much more readily obtained. Single crystals of complexes **3.10** and **3.11** that were suitable for X-ray structure determination were grown by vapour diffusion of diethyl ether into MeOH solutions of the complexes. Structures of complexes **3.10** and **3.11** are described later in this chapter.

A further pair of 4'-(4-toluyl)-2,2':6',2''-terpyridine (ttp) based complexes, **3.13** and **3.14**, have also been prepared (Scheme 3.3) and crystallographically characterised.



Scheme 3.3. (i) EtOH: water (1: 4), reflux, 5 hr; (ii) LiCl, reflux, 30 min; (iii) NH_4PF_6; (iv) bpe, EtOH: water (1: 1), reflux, 5 hr; (v) NH_4PF_6.

The ttp complexes were prepared as shown in Scheme 3.3. These complexes are new compounds, but they were prepared using reaction conditions that are very similar to those used for the closely related terpyridine complexes.[398-400] Reaction of ruthenium complex **3.2** with bpy in boiling aqueous EtOH in the presence of LiCl afforded the crude complex **3.13**. After purification on silica gel, the product was collected as its PF_6^- salt by the addition of excess NH_4PF_6. ESI-MS of the red powder in CH_3CN solution reveals a signal at *m/z* 616.29 that can be assigned to the $[Ru(ttp)(bpy)Cl]^+$ ion. The observed isotope patterns are a close match to the calculated isotopic distribution patterns for this species.

Reaction of complex **3.13** with excess *trans*-1,2-bis(4-pyridyl)ethylene (bpe) ligand in aqueous EtOH gave complex **3.14** in good yield (70%). Purification of the crude material was again achieved on silica (Figure 3.4). The last major fraction was isolated as its PF_6^- salt, a red-orange powder. Again, the complex was characterised by NMR (see Scheme 3.3 for NMR numbering) and ESI-MS techniques. ESI-MS isotope patterns at *m/z* 908.43 and 381.63, can be assigned to $\{[Ru(ttp)(bpy)(bpe)](PF_6)\}^+$ and $[Ru(ttp)(bpy)(bpe)]^{2+}$, respectively (Figure 3.5). The observed isotope patterns are also a close match to the calculated isotopic distribution patterns.

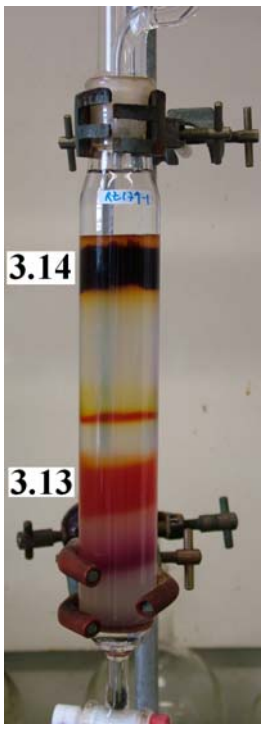


Figure 3.4. Well separated bands on a silica gel column chromatogram. The first orange band was the unreacted starting material, **3.13**, and the last major red band was the desired product, **3.14**.

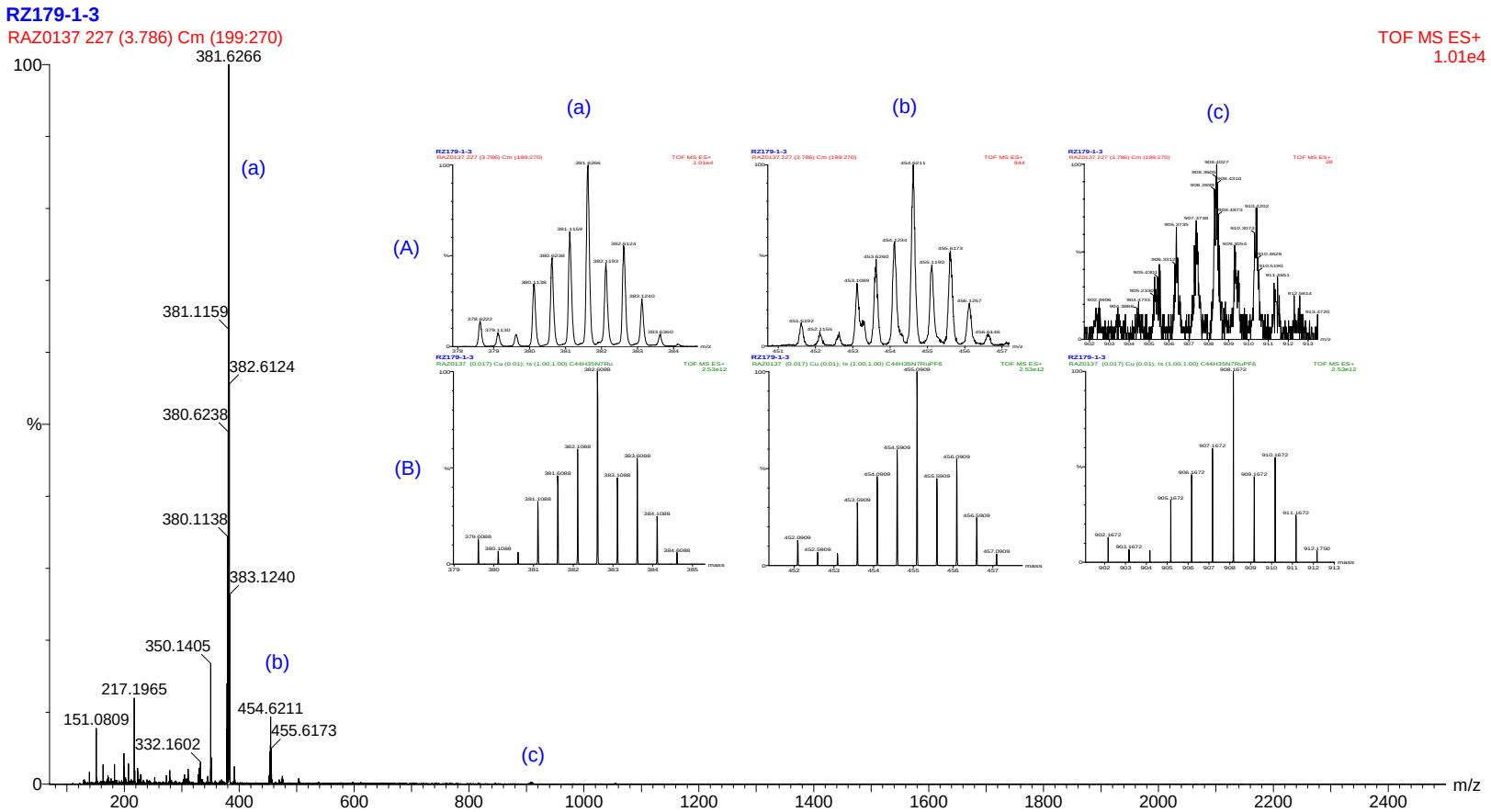


Figure 3.5. ESI-MS of $[Ru(ttp)(bpy)(bpe)](PF_6)_2$, **3.14**, in CH_3CN solution. (A) High resolution scans of the three major signals (a), (b), and (c); (B) Calculated isotope distribution patterns for (a) $[Ru(ttp)(bpy)(bpe)]^{2+}$, (b) $\{[Ru(ttp)(bpy)(bpe)](PF_6)+H\}^{2+}$, and (c) $\{[Ru(ttp)(bpy)(bpe)](PF_6)\}^{+}$, at *m/z* 381.63, 454.62, and 908.43, respectively.

3.3. X-Ray Crystallography

3.3.1. Crystal Structures of [Ru(tpm)(bpy)Cl](PF$_6$), 3.10, and [Ru(tpm)(bpy)(H$_2$O)](ClO$_4$)$_2$.MeOH, 3.11

Single crystals of **3.10** and **3.11** were grown by vapour diffusion of diethyl ether into the MeOH solutions of the complexes at room temperature. Perspective views of complexes **3.10** and **3.11** with the atom numbering are presented in Figures 3.6 and 3.7, respectively.

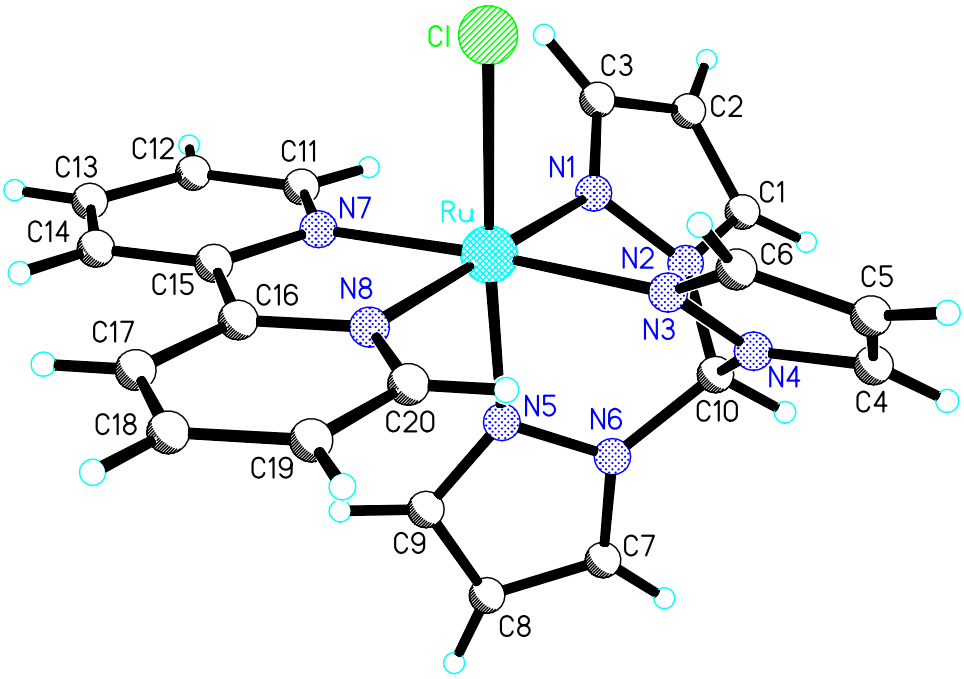


Figure 3.6 Structure of complex **3.10** with numbering scheme adopted. One hexafluorophosphate anion is omitted for clarity. Selected bond lengths [Å] and angles [°]: Ru-N(5) 2.022(2); Ru-N(7) 2.035(2); Ru-N(8) 2.044(2); Ru-N(3) 2.065(2); Ru-N(1) 2.063(2); Ru-Cl 2.4114(7); N(5)-Ru-N(7) 90.49(9); N(5)-Ru-

N(8) 92.12(9); N(7)-Ru-N(8) 79.05(9); N(5)-Ru-N(1) 85.88(9); N(7)-Ru-N(1) 99.01(9); N(8)-Ru-N(1) 177.22(9); N(5)-Ru-N(3) 87.64(9); N(7)-Ru-N(3) 176.24(9); N(8)-Ru-N(3) 97.74(9); N(1)-Ru-N(3) 84.12(9); N(5)-Ru-Cl 175.26(6); N(7)-Ru-Cl 91.49(7); N(8)-Ru-Cl 92.48(7); N(1)-Ru-Cl 89.56(7); N(3)-Ru-Cl 90.62(7).

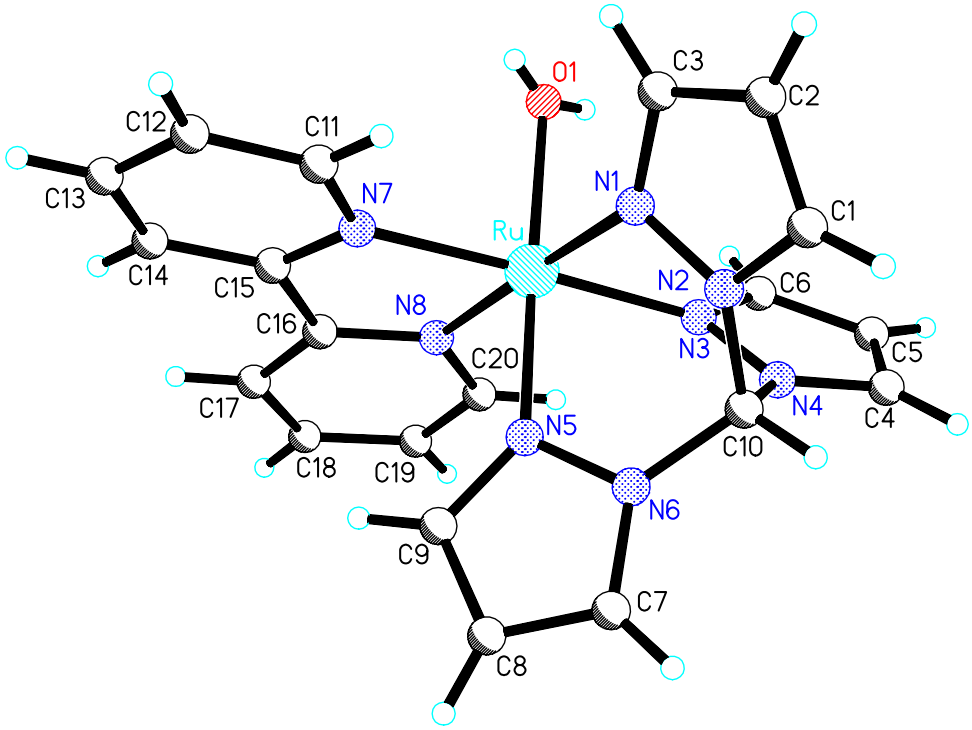


Figure 3.7. Structure of complex **3.11** with numbering scheme adopted. Two perchlorate anions and one solvated methanol molecule are omitted for clarity. Selected bond lengths [Å] and angles [°]: Ru-N(5) 2.011(3); Ru-N(7) 2.051(4); Ru-N(8) 2.053(3); Ru-N(3) 2.076(4); Ru-N(1) 2.077(4); Ru-O(1) 2.151(3); N(5)-Ru-N(7) 91.63(14); N(5)-Ru-N(8) 88.69(13); N(7)-Ru-N(8) 79.06(14); N(5)-Ru-N(1) 87.51(14); N(7)-Ru-N(1) 99.63(14); N(8)-Ru-N(1) 175.94(14); N(5)-Ru-N(3) 87.49(13); N(7)-Ru-N(3) 177.87(14); N(8)-Ru-N(3) 98.97(14); N(3)-Ru-

N(1) 82.28(13); N(5)-Ru-O(1) 178.29(13); N(7)-Ru-O(1) 89.22(13); N(8)-Ru-O(1) 92.93(13); N(1)-Ru-O(1) 90.89(13); N(3)-Ru-O(1) 91.70(13).

In both structures **3.10** and **3.11**, the Ru(II) ions adopt an approximately octahedral geometry with three N atoms (N1, N2, and N5) from tpm ligands coordinated in a facial fashion. Planar bidentate bpy ligands occupy two other positions (N7 and N8), and the sixth coordination site is occupied by a chloride anion in **3.10** or a water molecule in **3.11**. Bond lengths and bond angles are within the range found for similar structures previously described in the literature.[385,397,399] The bond length to the pyrazolyl donor that is *trans* to the monodentate ligand is shorter than those to the other two donors in both complexes. This may be due to the relative *trans* influences of the non-tpm ligands in these complexes. However, the bond angle between the pyrazole donors *trans* to the bpy ligand is marginally smaller than the other angles subtended at ruthenium by the facial tpm ligand. This may be due to steric clashes with the bpy ligand, and provides an alternative explanation for the different bond lengths to the pyrazole donors.

There are π-π stacking interactions between the planes of bpy ligands of the complexes (face-face) within the lattices of structures **3.10** and **3.11**. The separations between the plane of bpy ligand of one molecule and the bpy ligand of the adjacent molecule in structures **3.10** and **3.11** are 3.4 and 3.5 Å, respectively (Figure 3.8 and Figure 3.9). The water ligand in structure **3.11** is involved in a hydrogen bonding network that involves the methanol solvent molecules and perchlorate anions in the lattice.

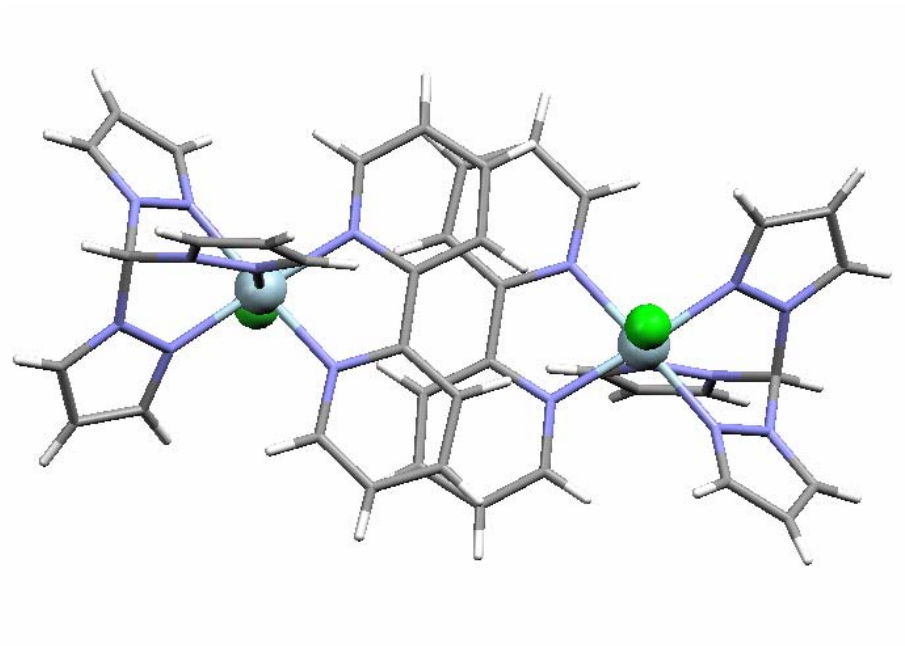


Figure 3.8. π-π stacking interactions between two adjacent molecule in structure **3.10**. The distance between the planes of bpy ligands in the adjacent cations is 3.4 Å.

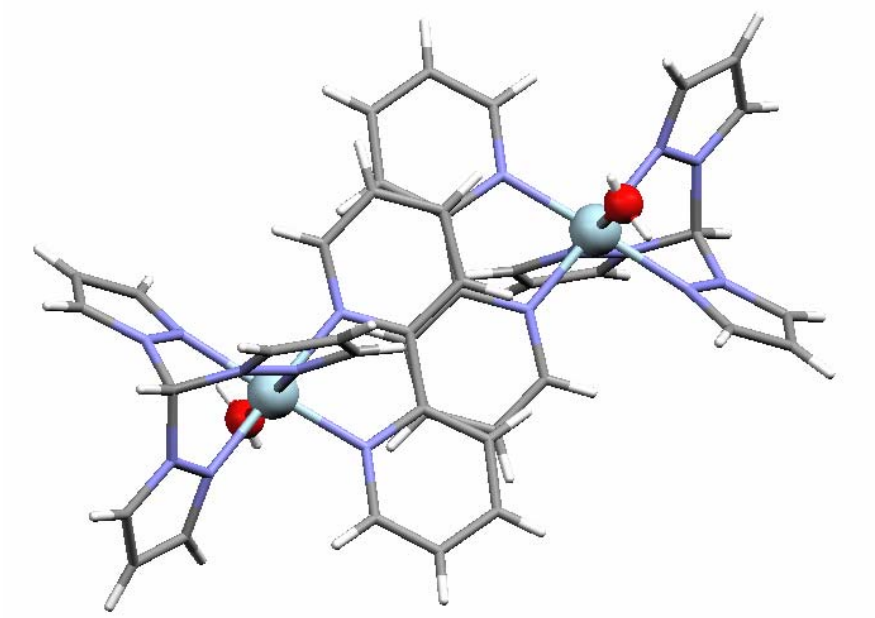


Figure 3.9. π-π stacking interactions between two adjacent molecule in structure **3.11**. The distance between the planes of bpy ligands in the adjacent cations is 3.5 Å.

The Ru-Cl bond length in **3.10** is 2.4114(7) Å, and very similar to that in the ttp complex, **3.13**, (Figure 3.10) at 2.4103(9) Å, while the Ru-OH_2 distance in **3.11** is 2.151(3) Å. In similar pairs of structures described in the literature, the Ru-Cl bond distances (2.395,[401] 2.431,[402] 2.387,[399] and 2.408 Å[403]) of the chloro complexes are also longer than Ru-OH_2 distances (2.127,[404] 2.126,[402] 2.119,[399] and 2.139 Å[403]) in the aqua complexes.

3.3.2. Crystal Structures of [Ru(ttp)(bpy)Cl](PF_6), 3.13, and [Ru(ttp)(bpy)(bpe)](PF_6)$_2$.MeOH, 3.14

Single crystals of **3.13** and **3.14** were obtained by vapour diffusion of diethyl ether into CH_3CN and CH_3CN/MeOH solutions of the complexes at r.t, respectively. Single crystals of each compound were used for structure determination. The structures of the cations are shown in Figures 3.10 and 3.11, respectively. The bond lengths and angles are within the range found for similar structures described in the literature.[399,401-403,405-413] In structures **3.13** and **3.14**, the shortest Ru-N bond length is the Ru-N bond to the central pyridine ring in ttp ligand. The Ru-N bond distances in bpy ligands which are *trans* to the monodentate ligands (Cl or bpe) are shorter than those in the other pyridine rings in bpy ligands. These observations are also entirely consistent with the literature values.[399,401-403,405-413]

As also shown in Figure 3.12, the structure is stabilized by π-π stacking interactions between the ttp planes. There are two types of π-π stacking interactions by which the lattice is stabilized. The distance between the planes of the flanking pyridine rings of the adjacent cations is 3.7 Å (centroid-centroid) and the separation of the

central pyridine plane in one cation to the flanking pyridine plane in the adjacent cation is approximately 3.6 Å (centroid-centroid).

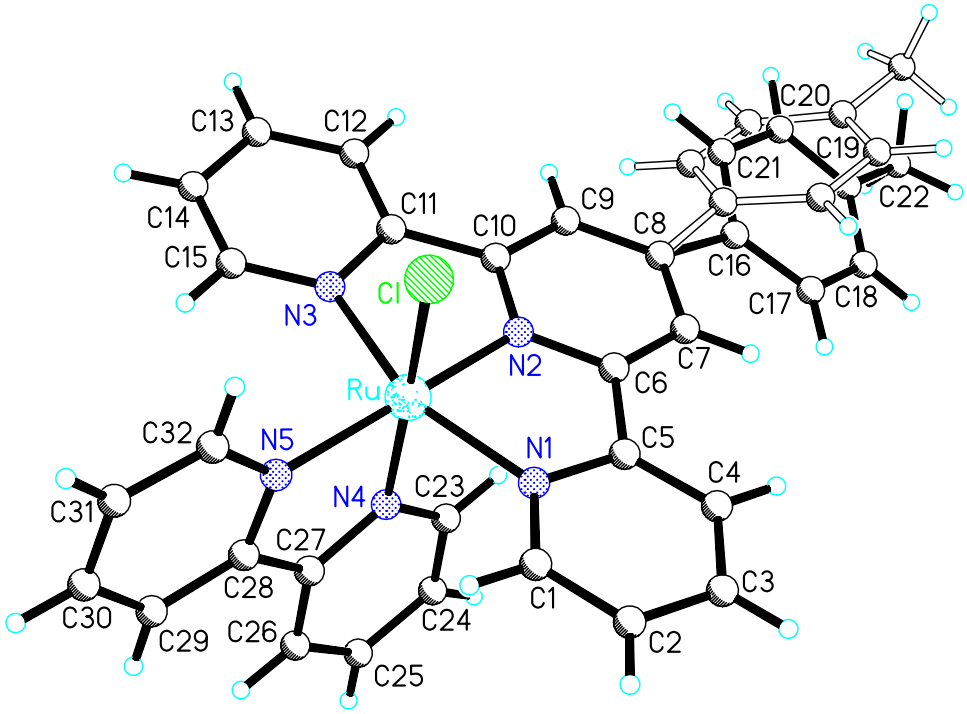


Figure 3.10. The molecular structure of complex **3.13**, with a PF_6^- anion omitted for clarity. The methylphenyl group of the ttp exhibits a disorder. Selected bond lengths [Å] and angles [°]: Ru-N(2) 1.949(3); Ru-N(4) 2.033(3); Ru-N(1) 2.064(3); Ru-N(3) 2.065(3); Ru-N(5) 2.079(3); Ru-Cl 2.4103(9); N(2)-Ru-N(4) 95.56(11); N(2)-Ru-N(1) 79.24(10); N(4)-Ru-N(1) 88.47(10); N(2)-Ru-N(3) 80.05(11); N(4)-Ru-N(3) 95.71(11); N(1)-Ru-N(3) 159.17(11); N(2)-Ru-N(5) 172.73(11); N(4)-Ru-N(5) 78.44(11); N(1)-Ru-N(5) 104.48(11); N(3)-Ru-N(5) 96.35(11); N(2)-Ru-Cl 91.63(8); N(4)-Ru-Cl 171.24(8); N(1)-Ru-Cl 87.96(7); N(3)-Ru-Cl 90.45(8); N(5)-Ru-Cl 94.73(8).

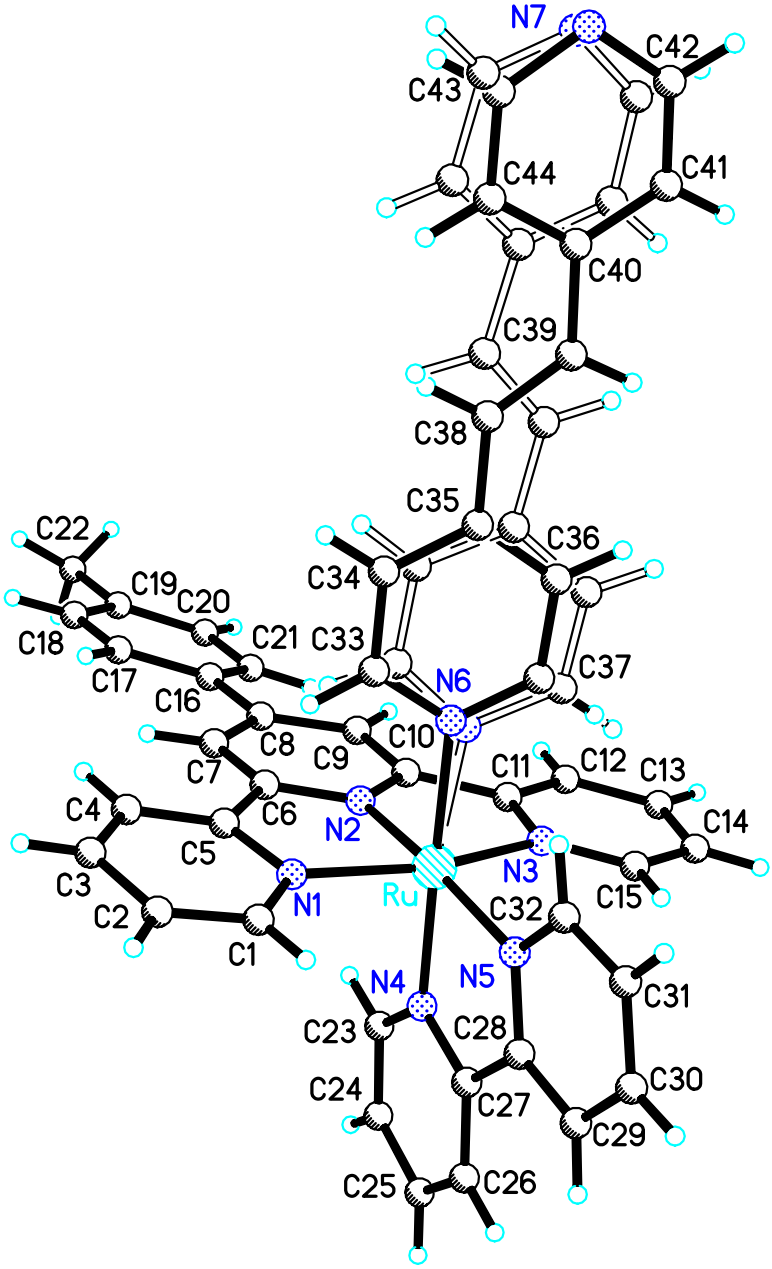


Figure 3.11. Molecular structure of complex **3.14**, with two hexafluorophosphate anions and a methanol molecule omitted for clarity. The coordinated bpe ligand exhibits pseudo 2-fold rotational disorder. One of the hexafluorophosphate ions is also disordered. Selected bond lengths [Å] and angles [°]: Ru-N(2) 1.955(5); Ru-

N(4) 2.056(5); Ru-N(1) 2.068(5); Ru-N(3) 2.067(5); Ru-N(5) 2.078(5); Ru-N(6) 2.10(4); N(2)-Ru-N(4) 97.2(2); N(2)-Ru-N(1) 79.6(2); N(4)-Ru-N(1) 91.7(2); N(2)-Ru-N(3) 79.6(2); N(4)-Ru-N(3) 88.3(2); N(1)-Ru-N(3) 159.0(2); N(2)-Ru-N(5) 175.7(2); N(4)-Ru-N(5) 78.5(2); N(1)-Ru-N(5) 100.1(2); N(3)-Ru-N(5) 100.4(2); N(2)-Ru-N(6) 89.1(18); N(4)-Ru-N(6) 172.5(15); N(1)-Ru-N(6) 93.3(13); N(3)-Ru-N(6) 88.9(15); N(5)-Ru-N(6) 95.2(18).

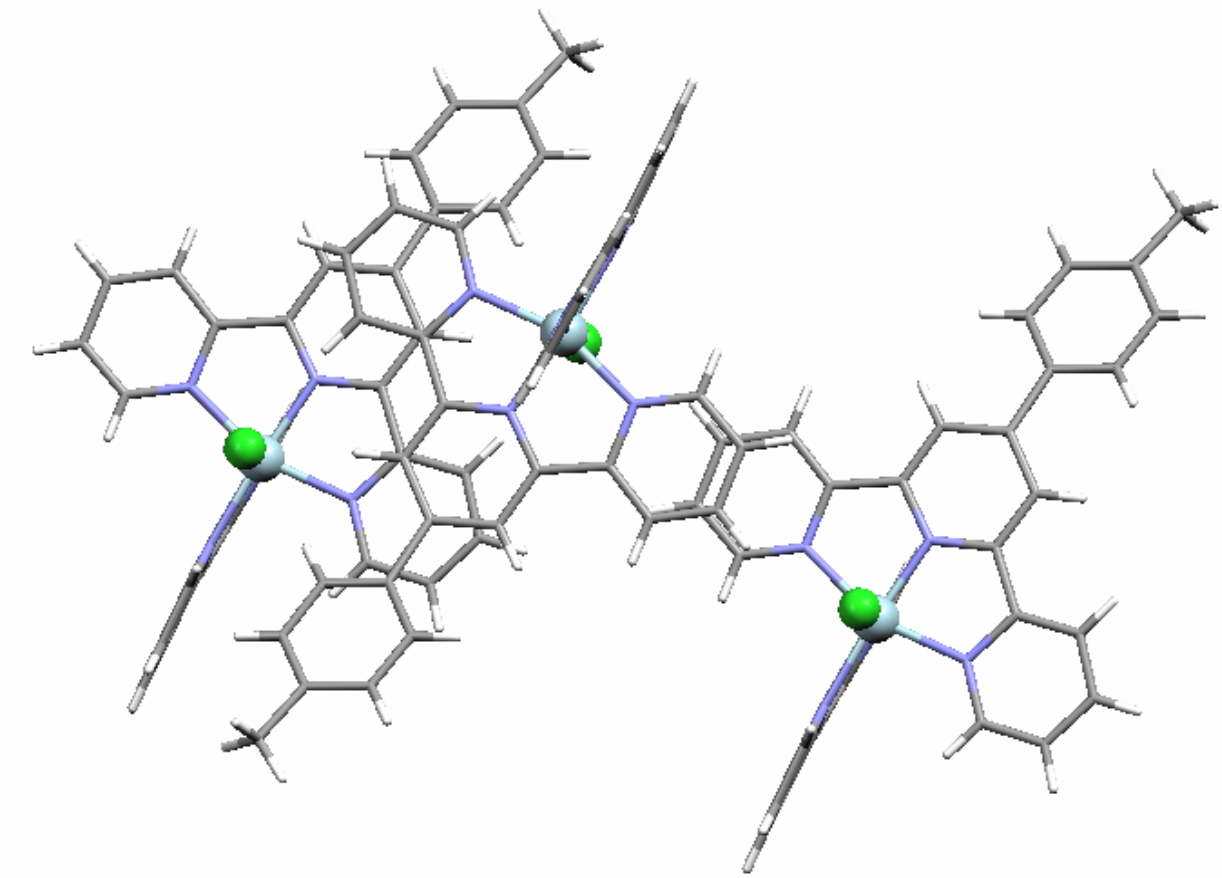


Figure 3.12. π-π stacking interactions (face-face) between the planes of the ttp groups in crystal structure of complex **3.13**. The distance between the planes of the flanking pyridine rings of the adjacent cations is 3.7 Å (centroid-centroid) and the separation of the central pyridine plane in one cation to the flanking pyridine plane in the adjacent cation is approximately 3.6 Å (centroid-centroid).

The structure of **3.14** reveals the bpe ligand to be disordered over two orientations in the solid state. The bond angles and distances are consistent with those of similar structures.[399,400] There are π-π stacking interactions between the bpe ligands of the adjacent complexes in the lattice of **3.14**. The separation between the plane of the coordinated pyridine ring of bpe in one cation to the plane of the uncoordinated pyridine ring in the adjacent cation is 3.7 Å (centroid-centroid). (Figure 3.13).

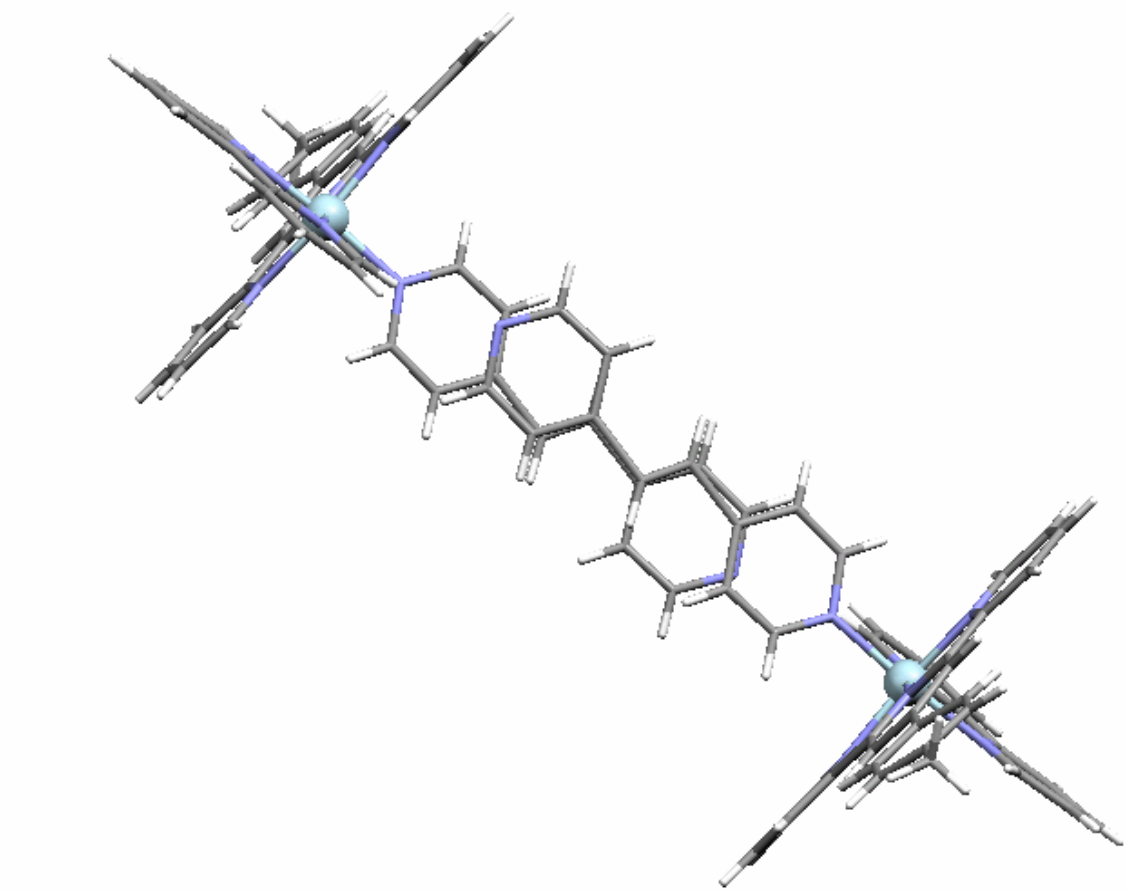


Figure 3.13. The π-π stacking interactions in the X-ray structure of complex **3.14**. The distances between the planes of the coordinated pyridine ring of bpe in one complex and uncoordinated pyridine ring in the adjacent cation is 3.7 Å (centroid-centroid).

3.4. Conclusion

Ruthenium(II) complexes of the ligands tris(1*H*-pyrazol-1-yl)methane (tpm), **3.1**, and 4'-(4-tolyl)-2,2':6',2''-terpyridine (ttp), **1.48**, have been prepared and studied as part of a model study for work with a more complicated and less readily available ditopic ligand 4'-(4-(2,2,2-tris(1*H*-pyrazol-1-yl)ethoxymethyl)phenyl)-2,2':6',2''-terpyridine (pzt), **1.71** (Figure 1.31 in Chapter 1). The ligand **1.71** was initially considered to link a Ru(II) ion at its terpyridine site and a Co(III) ion at its pyrazolyl binding domain. To the best of our knowledge, there is no report of Co(III) complexes of tpm complexes in the literature. This study, therefore, was undertaken to evaluate the possibility of employing the tpm portion of **1.71** to bind the Ru(II) ion with the terpyridine site being reserved for the Co(III) ion. This would involve using the $[Ru(tpm)Cl_3]$ complex in the reaction with **1.71**, with the idea of restricting coordination to the tpm binding site.

In $[Ru(tpm)_2Cl]^+$, one tpm ligand is found to coordinate in a facial tridentate manner, while the second tpm ligand acts as a bidentate ligand. The pendant pyrazolyl group of this second tpm ligand could not be induced to coordinate, even when the chloride ligand is removed by reaction with silver(I) ions. This means that regioselectivity of Ru coordination to **1.71** may be difficult to achieve when the tpm site, acting as a bidentate donor, is competing with the terpyridine site that could also act as a bidentate ligand.

It is well established in the literature[414] that polypyridyl Ru(II) complexes containing non-polypyridyl ancillary ligands, such as $[Ru(tpm)_2Cl]^+$ in our case, do not exhibit the outstanding excited state properties of the parent polypyridyl complex. Therefore, we replaced the chloride ion in $[Ru(tpm)_2Cl]^+$ cation with a strong field non-chromophoric ligand, CH_3CN, for our studies. We, then, wished to extend this

synthesis methodology to the bridging ligand **1.71** to prepare a dinuclear Ru(II)-Co(III) complex in which the Ru(II) ion is bound to the tpm portion of the ligand, and the Co(III) ion is linked to the terpyridyl site of the ligand. The Ru(II) ion is also coordinated to a tpm ligand, and a CH_3CN molecule for the photochemical studies, and the Co(III) ion is bound to a ligand which could be released upon irradiation by light. The coordination chemistry of the ditopic ligand **1.71** will be discussed later in Chapter 4.

In this Chapter, X-ray crystallographic studies were also reported for four compounds: [Ru(tpm)(bpy)Cl](PF_6), **3.9**, [Ru(tpm)(bpy)(OH_2)](ClO_4)$_2$, **3.10**, [Ru(ttp)(bpy)Cl](PF_6), **3.13** and [Ru(ttp)(bpy)(bpe)](PF_6)$_2$, **3.14** (bpe = *trans*-1,2-bis(4-pyridyl)ethylene).

Chapter 4

Coordination Chemistry of a New Terpyridine-Tris(pyrazolyl) Ditopic Ligand (4'-(4-(2,2,2-Tris(1H-pyrazol-1-yl)ethoxymethyl)phenyl)-2,2':6',2"-terpyridine)

4.1. Introduction

In this chapter, the focus is on the preparation and use of a new ditopic ligand where the two metal ion binding sites are differentiated by the configurations of the binding sites and the types of donor atoms that are present in the sites.

The ligand, 4'-(4-(2,2,2-tris(1*H*-pyrazol-1-yl)ethoxymethyl)phenyl)-2,2':6',2"-terpyridine (pzt)[348], **1.71** (Figure 4.1), contains a terpyridine fragment and a tris(pyrazolyl) fragment, which may allow synthetic differentiation between the tridentate binding sites as a result of the meridional and the facial coordination preferences of these groups around the octahedral metal centres.

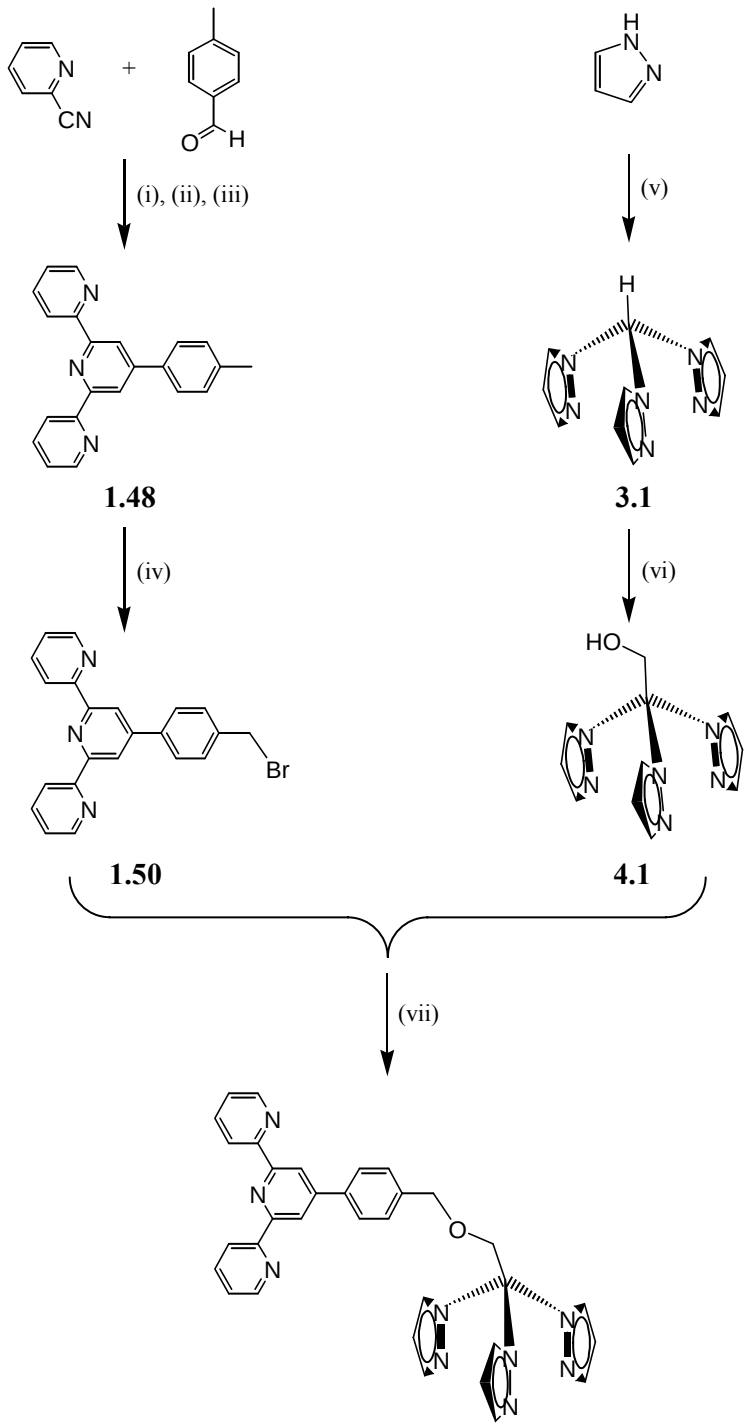
This designed bridging ligand may offer the benefits of polydentate ligands and minimisation of isomeric possibilities as was described in Chapter 1.

The tris(pyrazolyl)methane fragment is of interest, because the coordination chemistry of such systems has also been studied.[194,202,263-287,385,396,397,412,415-418] It, too, is easy to prepare, by reaction of pyrazole with chloroform, and to derivatise,[263,275] for example by reaction with formaldehyde, to give a molecule, 2,2,2-tris(1*H*-pyrazol-1-yl)ethanol,[263,275] that can be readily joined to the other binding site, a terpyridine containing synthon.

This chapter describes the synthesis, characterisation, and coordination chemistry of this new ditopic ligand. The free ligand and six metal complexes have been characterised by X-ray crystallographic techniques.

4.2. Synthesis of the Ligand

The ligand synthesis is relatively straightforward. Syntheses of suitable precursor molecules for both ends of the ditopic ligand are available in the literature. 4'-(4-(bromomethyl)phenyl)-2,2':6',2"-terpyridine, **1.50**,[217,218] is readily prepared by radical bromination of the related alkane, and this was reacted with 2,2,2-tris(1*H*-pyrazol-1-yl)ethanol, **4.1**,[263] in the presence of sodium hydride.



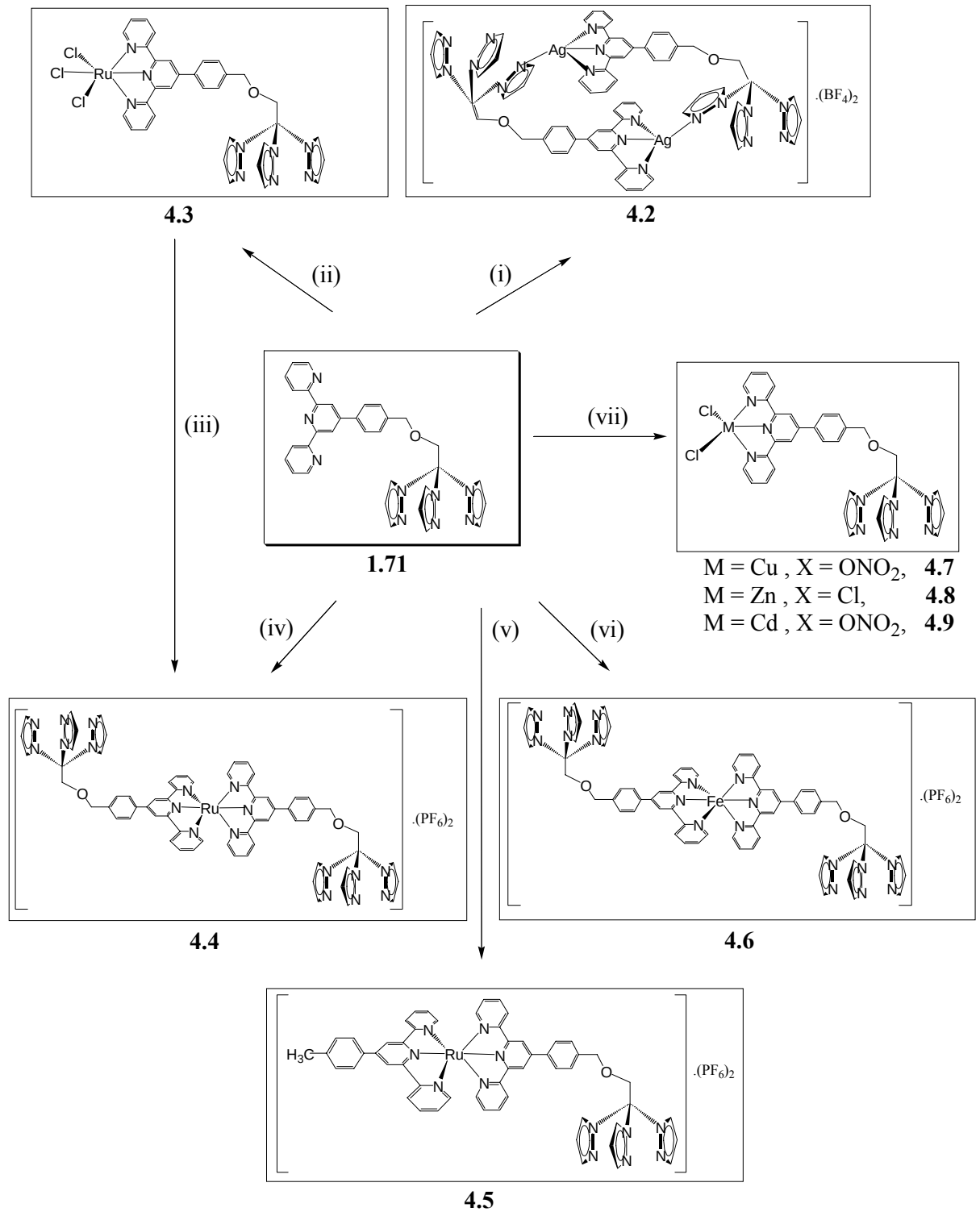
Scheme 4.1. Synthesis of ligand pzt. (i) $[NH_4][OAc]$, NaOH, acetamide, EtOH, reflux; (ii) $FeCl_2.4H_2O$, NH_4PF_6; (iii) H_2O_2; (iv) BPO, NBS, dry benzeze, *hυ*, reflux 4hr under Ar;(v) Na_2CO_3, $CHCl_3$, *tetra*-*N*-butylammonium bromide, reflux; (vi) potassium *tert*-butoxide, *para*-formaldehyde, reflux, under Ar; (vii) NaH, dry CH_3CN, reflux 24 hr, under Ar.

The resulting ligand, pzt, **1.71**, was purified by recrystallisation and has been fully characterised by NMR spectroscopy, elemental analysis, X-ray crystallography, and electrospray mass spectrometry (ESI-MS) techniques.

4.3. Syntheses and Characterisation of Some Metal Complexes of The 4'-(4-(2,2,2-Tris(1*H*-pyrazol-1-yl)ethoxymethyl)phenyl)-2,2':6',2''-terpyridine (pzt) Ligand

New metal complexes of the ligand pzt, **1.71**, were prepared upon reaction of the ligand **1.71** with Ru(II), Fe(II), Cu(II), Cd(II), Zn(II), and Ag(I) (Scheme 4.2).

Metal ions with a preference for octahedral geometry form ML_2 complexes that are readily isolated and characterised, with the metal ion being bound to the terpyridine sites of both ligands. Other metal ions bind to the terpyridine site of just one ligand. In the case of silver(I), a dinuclear M_2L_2 complex has been isolated in which each silver ion is coordinated to the terpyridine site of one ligand and to a single pyrazolyl donor group from the second ligand. Evidence for binding of metal ions to the tris(pyrazolyl) binding site was obtained by electrospray mass spectrometry and NMR techniques.



Scheme 4.2. Syntheses of transition metal-pzt complexes. (i) $AgBF_4$, CH_2Cl_2/CH_3CN (1:1), r.t overnight; (ii) $RuCl_3.3H_2O$, EtOH, reflux 4hr; (iii) 1. pzt (1 equimolar), *N*-methylmorpholine, MeOH, reflux 1 hr. 2. excess NH_4PF_4; (iv) 1. $RuCl_3.3H_2O$ (0.5 equimolar), *N*-methylmorpholine, MeOH, reflux 1 hr. 2. excess NH_4PF_4; (v) 1.

[Ru(ttp)Cl_3] (1 equimolar), *N*-methylmorpholine, MeOH, reflux 1 hr. 2. excess NH_4PF_4; (vi) 1. $FeCl_2.4H_2O$ (0.5 equimolar), CH_2Cl_2/EtOH, r.t, two hr. 2. excess NH_4PF_4; (vii) MX_2 ($Cu(NO_3)_2$ or $Cd(NO_3)_2$ or $ZnCl_2$) in appropriate solvents, r.t.

4.3.1. Silver Complexes

Many ligands that contain the tris(pyrazolyl)methane fragment have been prepared, characterised, and complexed with a range of metal ions.[194,202,253,262-274,283,284,396,416,417,419-423] The ability of silver(I) to adopt a wide range of coordination numbers and geometries has led to it being used in a significant number of such studies. Indeed, the work from Reger's group in particular,[266,271-273] has demonstrated the ability of silver(I) to form complexes of this class of ligand. Clearly then, silver is a good metal ion with which to begin the discussion of the coordination chemistry of the pzt ligand.

Reaction of the pzt ligand, dissolved in CH_2Cl_2, with $AgBF_4$, dissolved in acetonitrile, gave a clear solution which produced a white solid upon evaporation of the solvent. The ^{1}H NMR spectrum of the powder sample, in dilute dmso-d_6 solution, has resonances similar to that of the ligand, but the chemical shifts of the signals corresponding to the phenyl-terpyridine portion of the molecule were shifted significantly, which is consistent with coordination of silver at this site. No significant change was observed for the chemical shifts of the pyrazolyl protons, even when excess $AgBF_4$ was used (Figure 4.1).

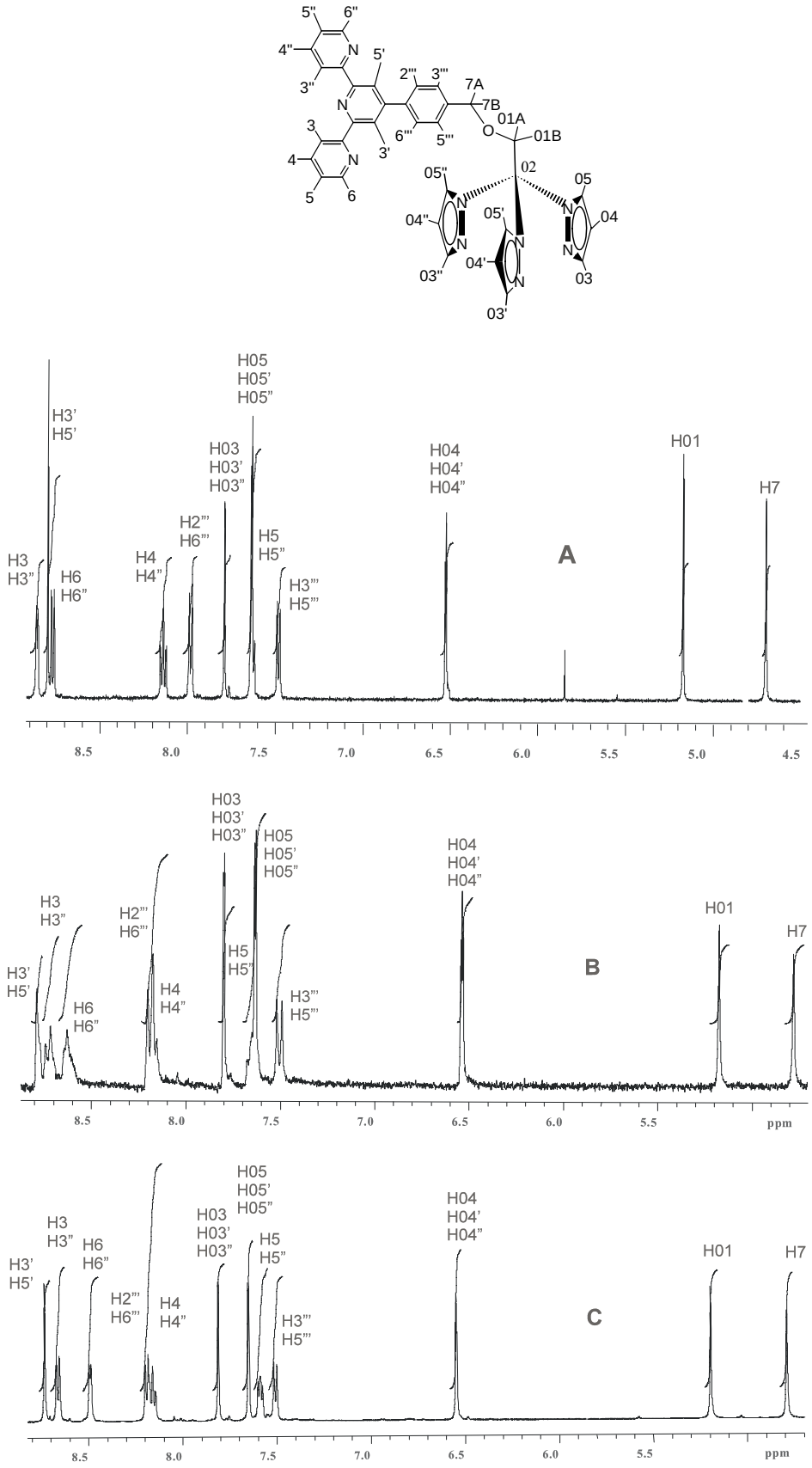


Figure 4.1. A. ^{1}H NMR spectrum of the pzt ligand in dmso-d_6. **B.** ^{1}H NMR spectrum of the dilute solution (0.002 M) of the white powder silver complex of

the ligand pzt as the BF_4^- salt in dmso-d_6. **C.** ^{1}H NMR spectrum of the concentrated solution (0.05 M) of the white powder silver complex of the ligand pzt as the BF_4^- salt in dmso-d_6.

Only one set of ligand signals is seen in the NMR spectra of solutions of ligand into which silver ions have been titrated, but the chemical shifts of the signals vary both with the quantity of silver ions that have been added (Figure 4.2) and with the concentration of the solution (Figure 4.3). These data are consistent with ligand exchange being fast on the NMR timescale. Furthermore, the changes in chemical shift are not continuous through the titrations, some resonances continue to move even after others have ceased to, and a large excess of silver ion is required to get to a point where significant changes in chemical shift cease. From these data we infer that there are multiple complex species present in solution at any one time. At high concentration, the signals assigned to the $H_{6,6''}$ and protons are observed at higher field than at lower concentration (Figure 4.3).

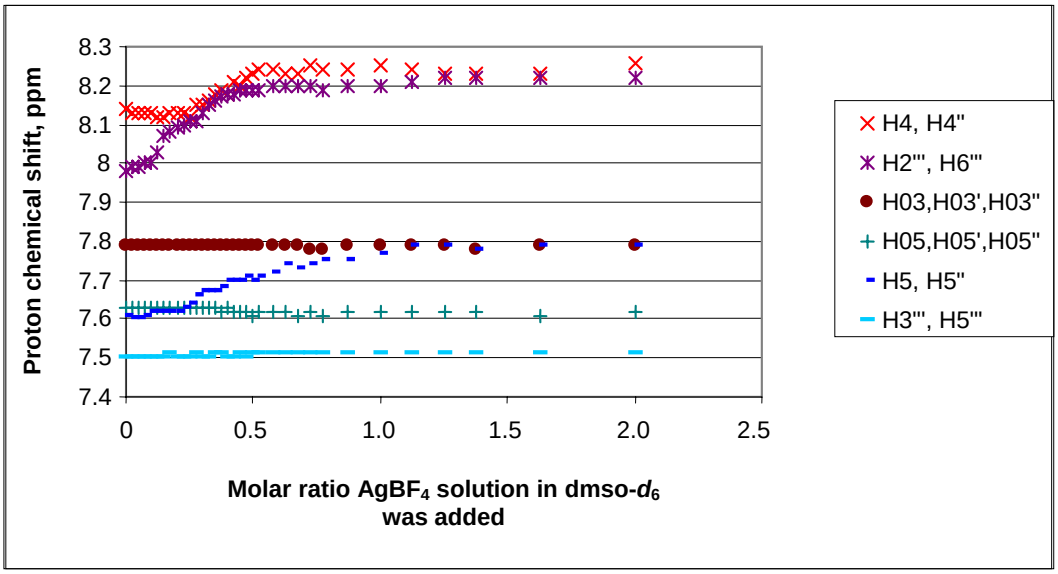


Figure 4.2. ^{1}H NMR titration of ligand pzt, **1.71**, with $AgBF_4$ in dmso-d_6 solution. (Concentrations were measured approximately).

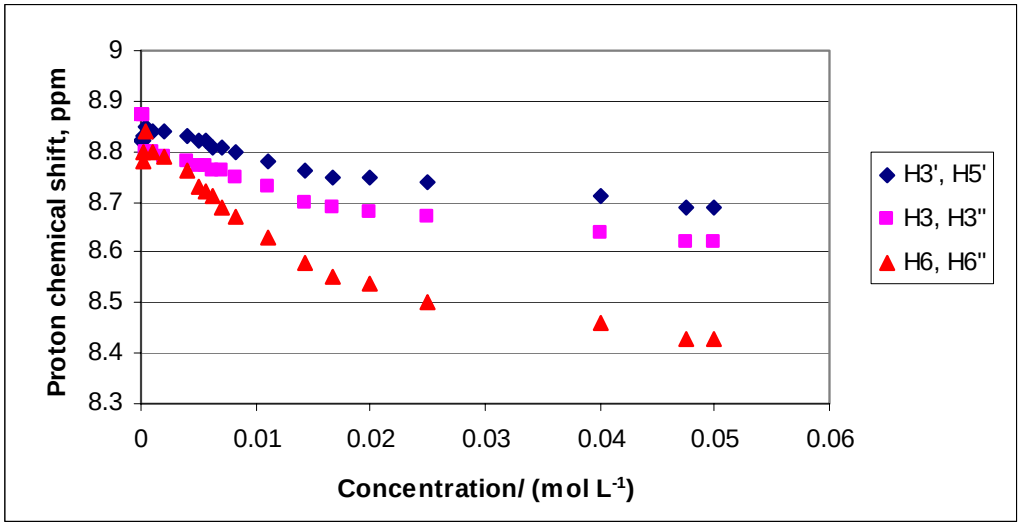


Figure 4.3. Plots of proton chemical shifts of the complex **4.2** in dmso-d_6 solutions with different concentrations.

ESI-MS of the white residue in CH_3CN solution shows mass to charge signals at 674.11, 1239.25, and 1433.15 units corresponding to $[2(pzt)+2Ag]^{2+}$, $[2(pzt)+Ag]^{+}$ and $[2(pzt)+2Ag+BF_4]^{+}$ ionic species, respectively. The observed isotope patterns are a close match to the calculated isotopic distribution patterns for ions of those compositions (Figure 4.5). It should be noted, however, that there are always large peaks due to the free ligand present in the mass spectrum (*m/z* 566.20 $[pztH]^{+}$), even if excess silver ion is present. Further, the signal intensities for the di-silver species increase as the silver:ligand ratio is increased (Figure 4.6).

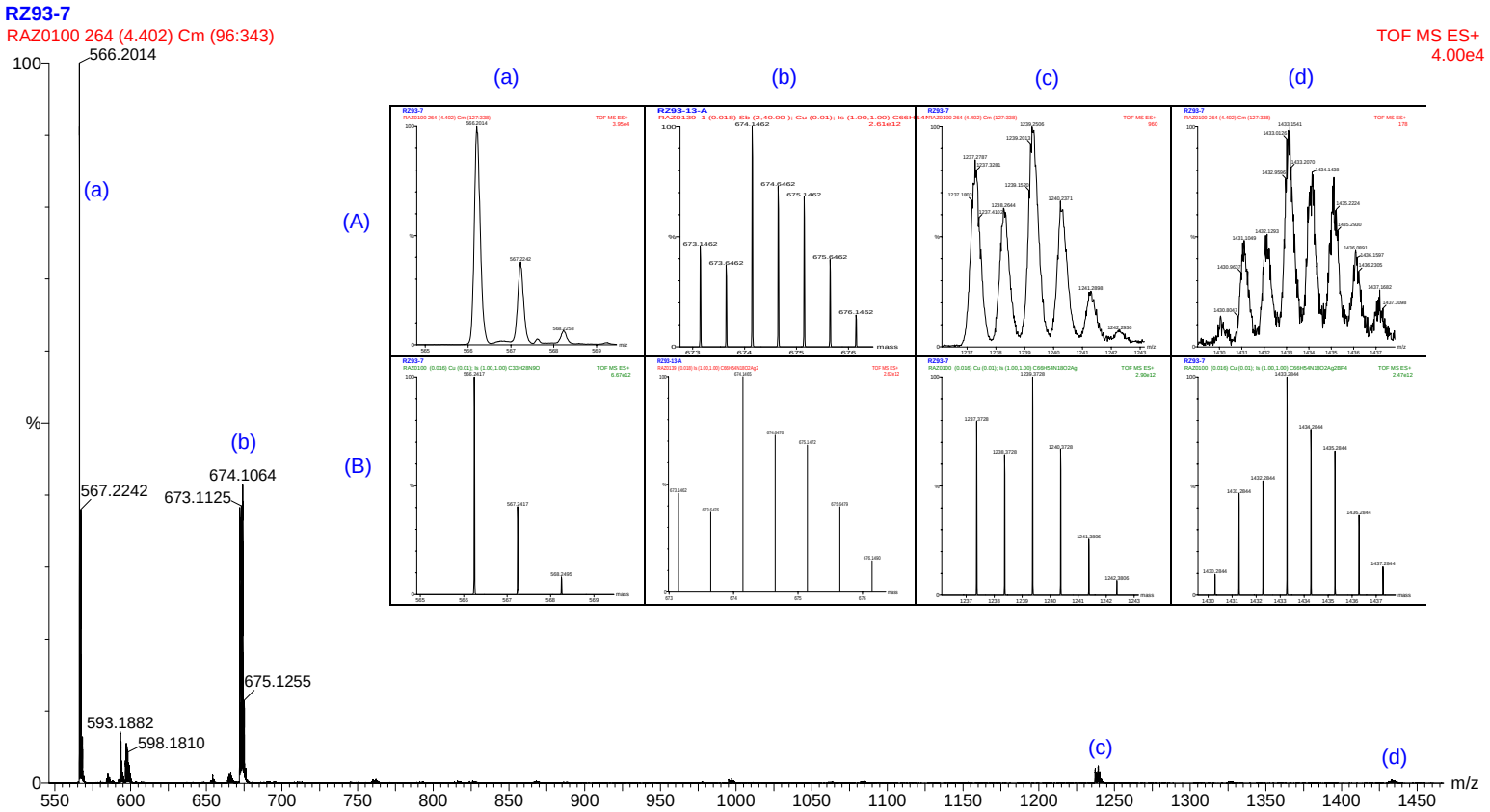


Figure 4.5. ESI-MS of material obtained from the reaction of ligand pzt with $AgBF_4$ in acetone. (A) High resolution scans of the four major signals (a)-(d); (B) Calculated isotope distribution patterns for (a) $[(pztH)]^+$, (b) $[Ag_2(pzt)_2]^{2+}$, (c) $[Ag(pzt)_2]^+$, (d) $[Ag_2(pzt)_2+(BF_4)]^{2+}$ at *m/z* 566.2, 674.11, 1239.25, and 1433.15 units, respectively.

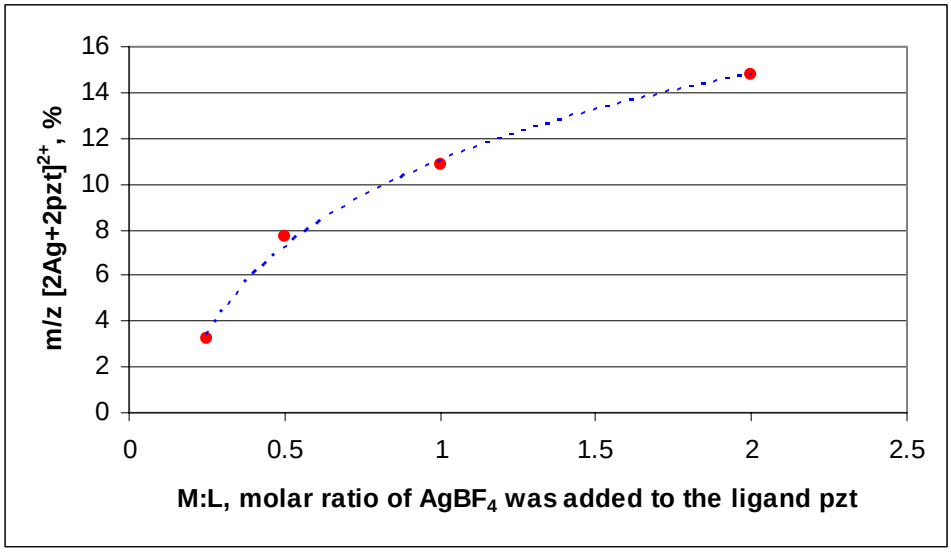


Figure 4.6. A plot of M:L, molar ratios of $AgBF_4$ were added to the ligand **1.71** against % of peak at *m/z* 674.1 corresponding to $[M_2(pzt)_2\text{-}2(BF_4)]^{2+}$ species.

The presence in the mass spectrum of complex ions in which more than one metal ion was present led us to conclude that, although the initial coordination was likely through the terpyridyl portion of the ligand (based on NMR data), the pyrazolyl portion of the ligand can also bind in some fashion to a silver ion. However, the very small changes that are seen in the ^1H NMR chemical shifts of signals assigned to the tris(pyrazolyl) portion of the molecule when silver ions are added to the solution may imply that the complexation of silver ions by the pyrazolyl portion of the ligand is significantly weaker than that at the terpyridyl site. Overall, these ESI-MS and NMR data imply that the silver complexes of the ligand are not particularly stable (at least in the coordinating solvents that we have employed), that they are certainly very labile, and that dinuclear systems are formed, particularly when silver ions and ligand are present in high concentration.

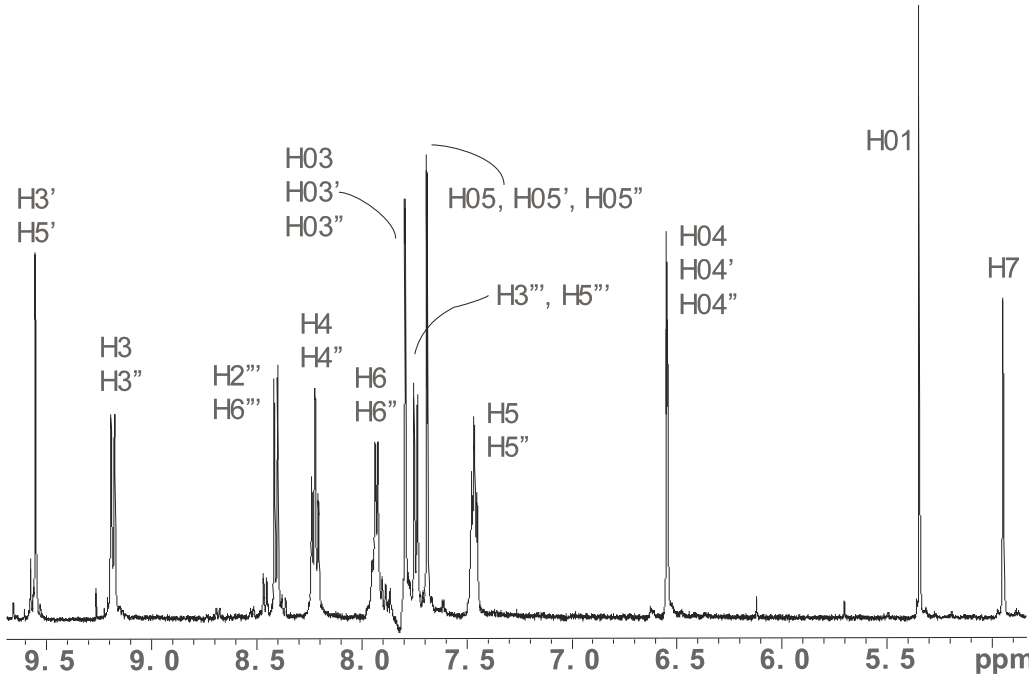
4.3.2. Ruthenium and Iron Complexes

As was already described in Chapter 1, terpyridyl ligand fragments are very good ligands for many different metal ions but the complexes of ruthenium(II)[143,156,221,230,231,236,238,244,385,424-429] and iron(II)[191,192,224,225,227,234,360,369,384,389,390,392,430-446] are probably among the most studied. As a part of our research studies, the pzt ligand has been used to prepare a number of new complexes, as shown in Scheme 4.2.

Following standard procedures for these kinds of systems, the neutral $[Ru(pzt)Cl_3]$ complex, **4.3**, can be isolated as an insoluble, dark brown, powder. This product was not further characterised. Ruthenium(III) species of this kind, [Ru(X-terpy)Cl_3], are extremely insoluble in most solvents, and their purification and further characterisation is not usually pursued.[188,231,388] The pzt ligand can be reacted with $[Ru(pzt)Cl_3]$, **4.3**, or with $[Ru(ttp)Cl_3]$, **3.2**, to give $[Ru(pzt)_2]^{2+}$, **4.4**, and $[Ru(pzt)(ttp)]^{2+}$, **4.5**, respectively. Alternatively, $[Ru(pzt)_2]^{2+}$, **4.4**, can be prepared directly, and was isolated in higher yield (79% *vs* 44%), when two equivalents of the pzt ligand were reacted with hydrated ruthenium trichloride in boiling MeOH.

For both $[Ru(pzt)_2]^{2+}$, **4.4**, and $[Ru(pzt)(ttp)]^{2+}$, **4.5**, the crude product was purified by column chromatography on silica. The major bands were collected and the products precipitated as the hexafluorophosphate salts. The compounds have been characterised by ^{1}H and ^{13}C NMR spectrometry, and the spectra fully assigned using COSY and 2D NOESY techniques (Figures 4.7, 4.8, 4.9, and 4.10). The chemical shift differences that are observed on comparison of the spectra for the complexes and the free ligand are entirely consistent with the expected coordination of the metal ion by the terpyridyl portion of the pzt ligand.

(A)



(B)

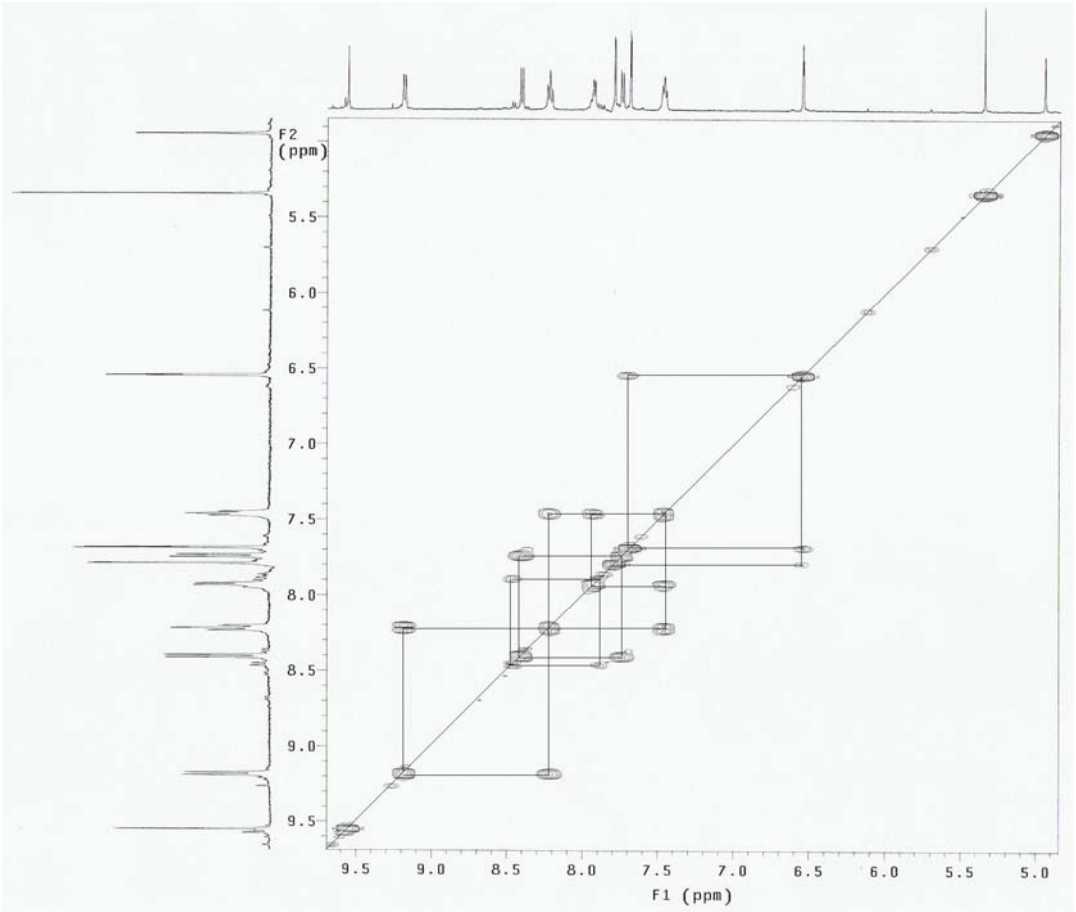


Figure 4.7. (A) ^{1}H NMR of the complex $[Ru(pzt)_2](PF_6)_2$, **4.4**, in acetone-d_6 solution. (B) ^{1}H-^{1}H COSY of the complex **4.4** in acetone-d_6 solution.

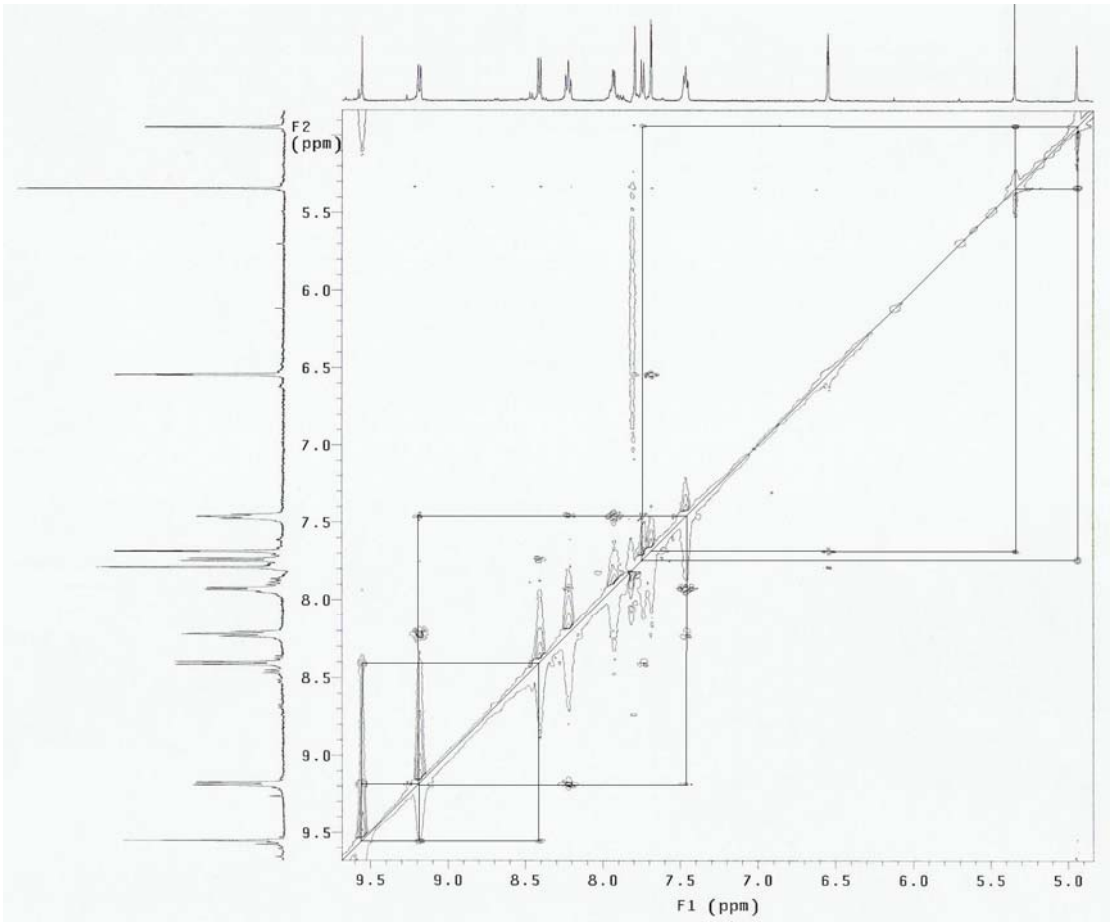
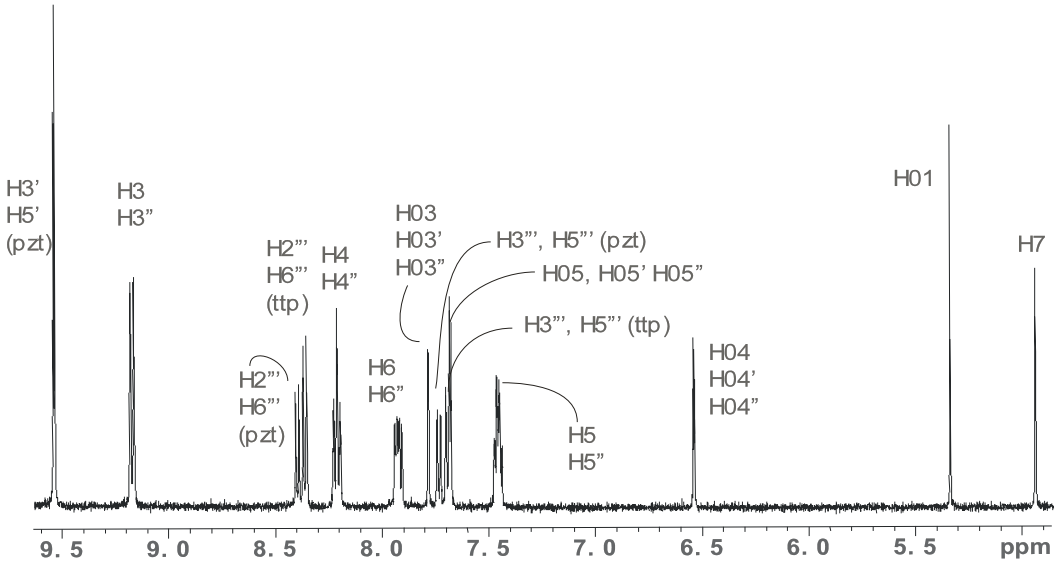


Figure 4.8. ^{1}H-^{1}H NOESY of the complex **4.4** in acetone-d_6 solution.

(A)



(B)

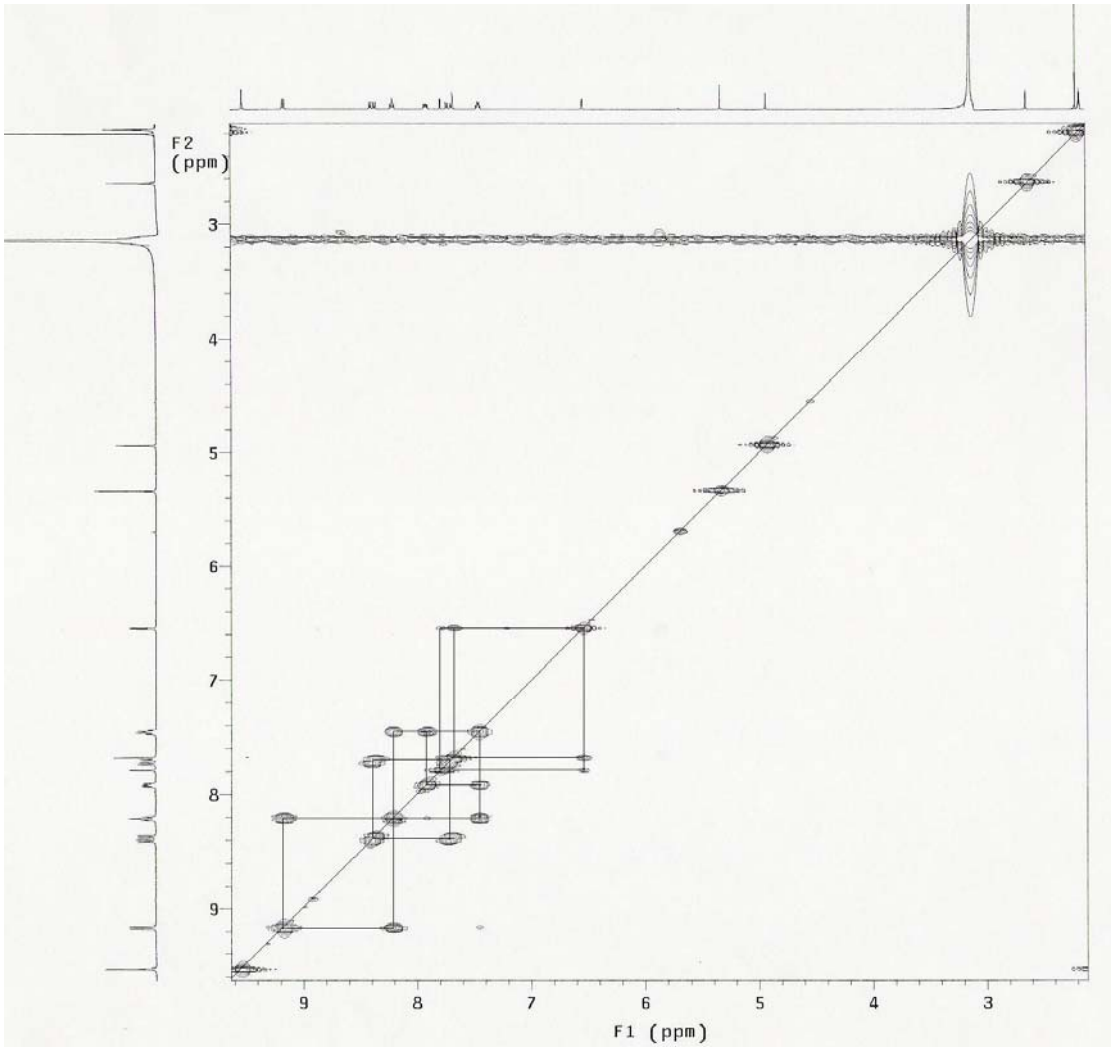


Figure 4.9. (A) ^{1}H NMR of the complex [Ru(ttp)(pzt)]$(PF_6)_2$, **4.5**, in acetone-d_6. (B) ^{1}H-^{1}H COSY of the complex **4.5** in acetone-d_6.

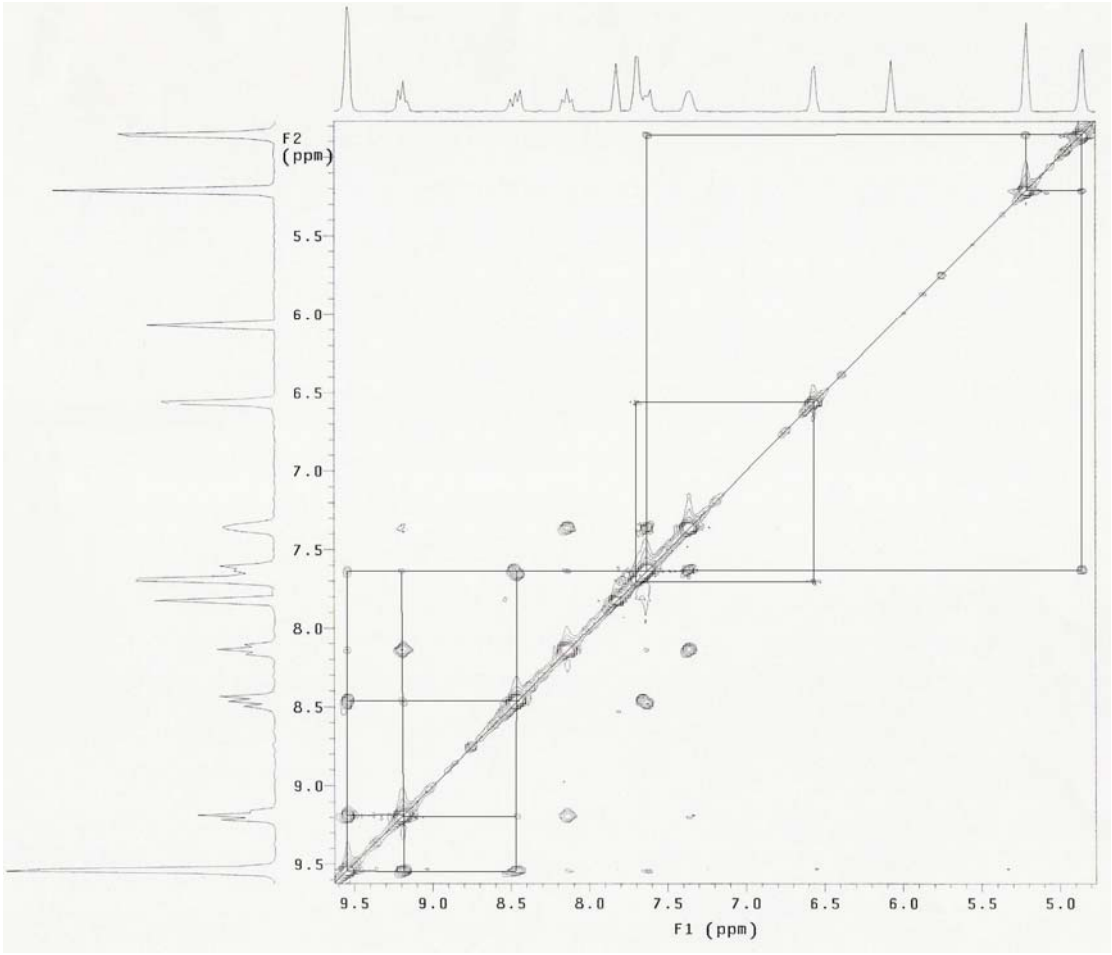


Figure 4.10. ^{1}H-^{1}H NOESY of the complex **4.5** in acetone-d_6.

The ESI-MS for the complexes exhibit peaks corresponding to $[M\text{-}PF_6]^+$ and $[M\text{-}2PF_6]^{2+}$ cations, at *m/z* 1377.6 and 616.3 for $[Ru(pzt)_2]^{2+}$ and 1135.5 and 495.2 for $[Ru(pzt)(ttp)]^{2+}$, with the observed isotope patterns correlating well with those predicted for the complex ions (Figures 4.11 and 4.12).

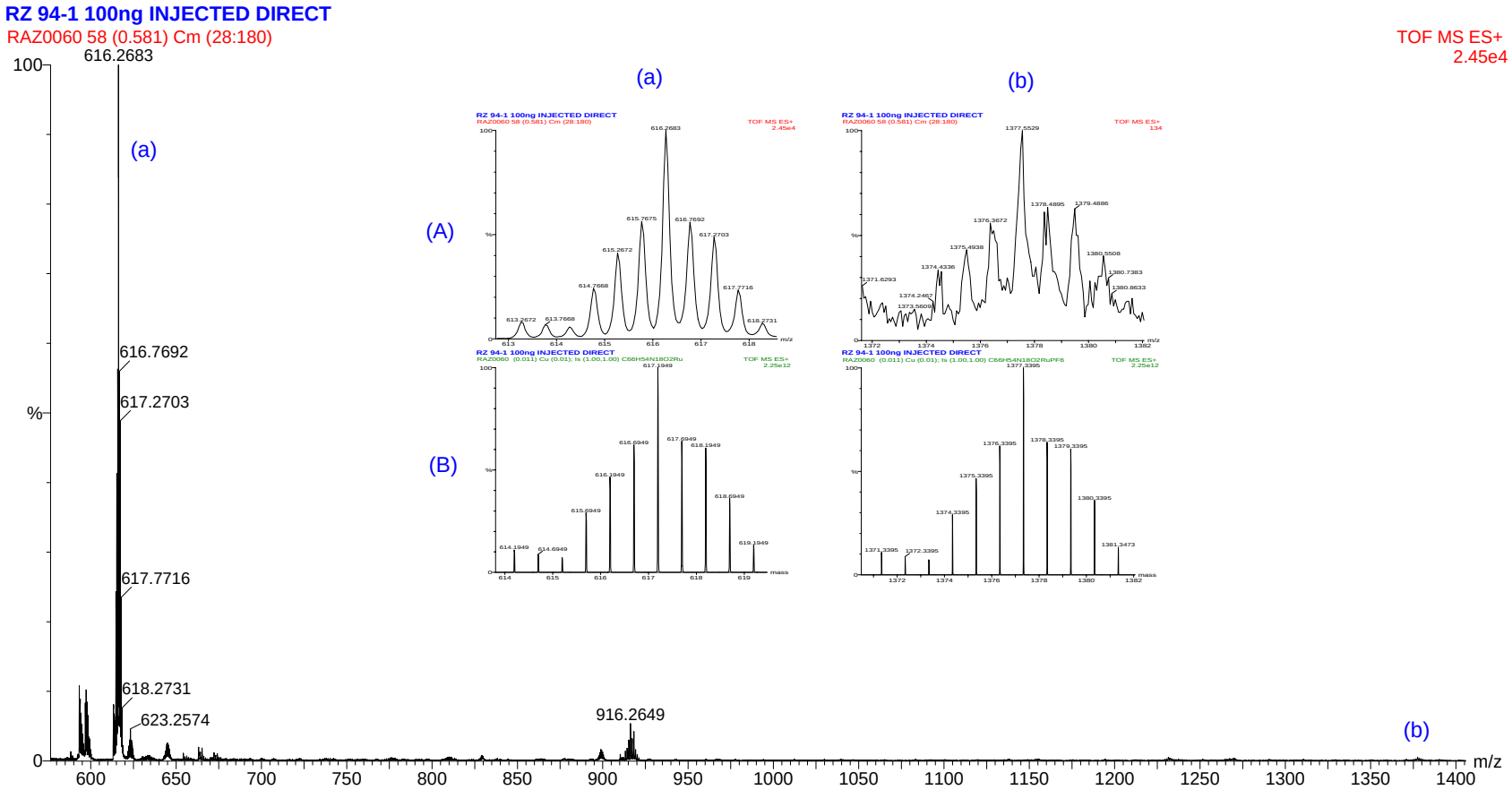


Figure 4.11. ESI-MS of $[Ru(pzt)_2](PF_6)_2$ complex, **4.4**, in CH_3CN. (A) High resolution scans of the two major signals (a) and (b); (B) Calculated isotope distribution patterns for (a) $[Ru(pzt)_2]^{2+}$ and (b) $[Ru(pzt)_2+(PF_6)]^{+}$ at *m/z* 616.3 and 1377.6, respectively.

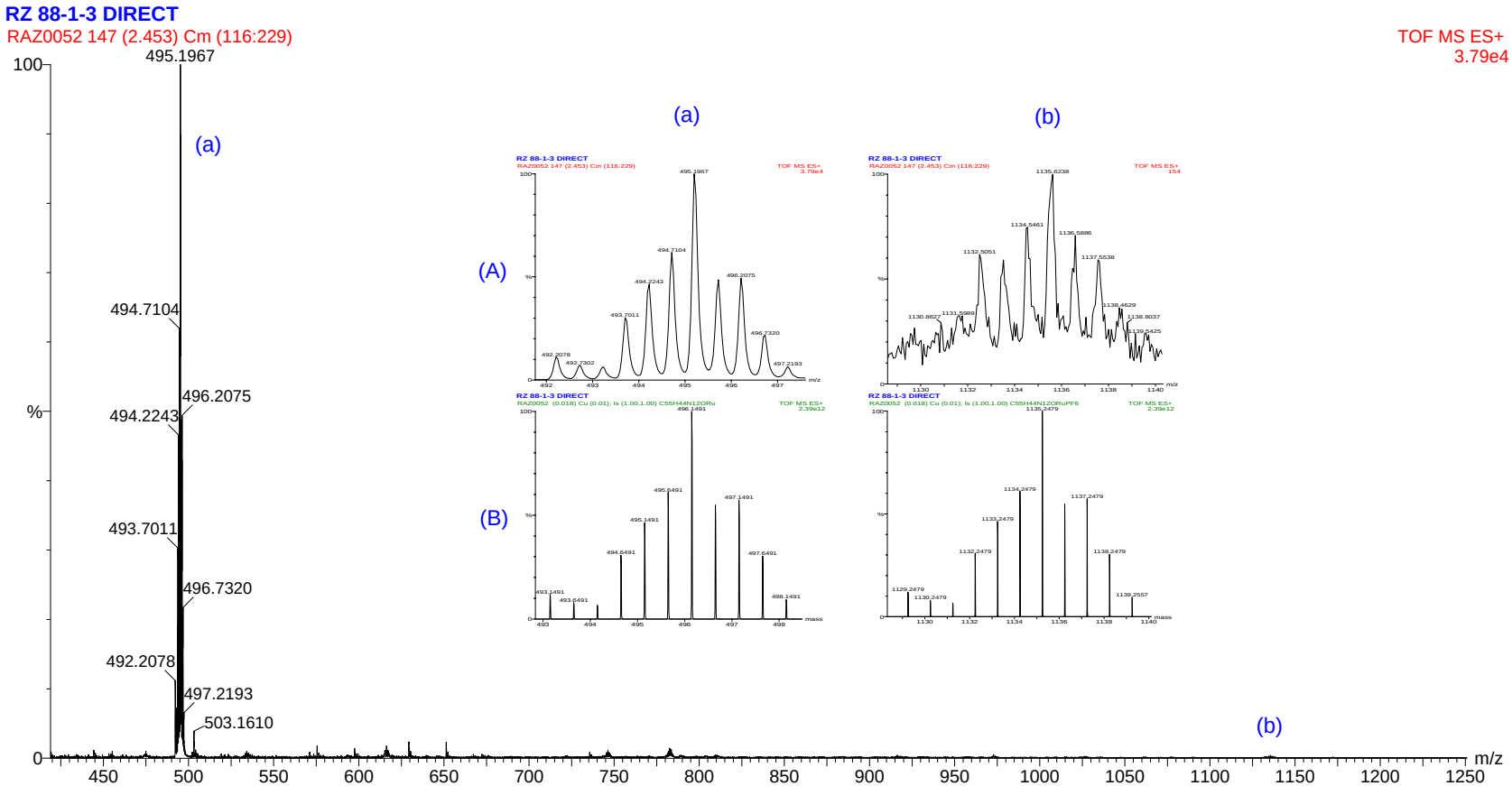
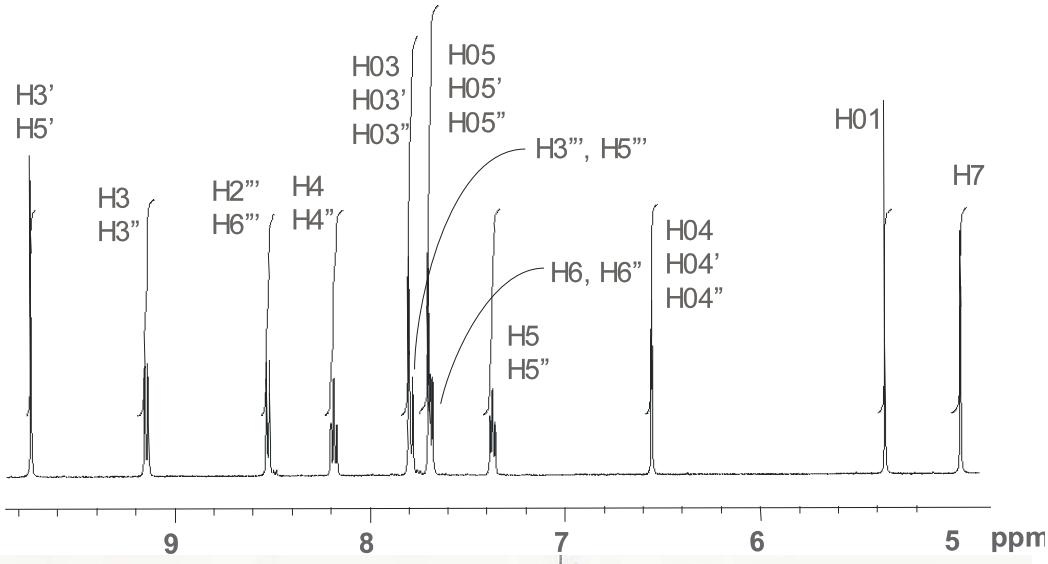


Figure 4.12. ESI-MS of complex **4.5** in CH_3CN. (A) High resolution scans of the two signals (a) and (b); (B) Calculated isotope distribution patterns for (a) $[Ru(pzt)(ttp)]^{2+}$ and (b) $[Ru(pzt)(ttp)+(PF_6)]^{+}$ at *m/z* 495.2 and 1135.5, respectively.

Mixing solutions of $FeCl_2.4H_2O$ (in EtOH) and pzt ligand (two equivalents, in CH_2Cl_2) resulted in the immediate formation of a dark-purple solution. The product $[Fe(pzt)_2](PF_6)_2$, **4.6**, was precipitated by addition an excess ammonium hexafluorophosphate to the solution, and further purification was achieved by recrystallisation from acetonitrile-water solution of the complex. Again, the NMR (Figures 4.13 and 4.14) and ESI-MS data (*m/z* 1331.6 and 593.2, corresponding to $[M-PF_6]^+$ and $[M-2PF_6]^{2+}$ (Figure 4.15)) are consistent with formulation of the complex as $[Fe(pzt)_2](PF_6)_2$, with coordination through the terpyridyl portion of the pzt ligand. In particular, ^1H NMR signals assigned to $H_{6,6''}$ and $H_{5,5''}$ (at 7.68 and 7.37 ppm respectively), are diagnostic for the complex containing two pzt ligands, as these signals are shifted up field from those in the free ligand as a result of the protons in one ligand being influenced by the magnetic field associated with the other ligand.[384]

(A)



(B)

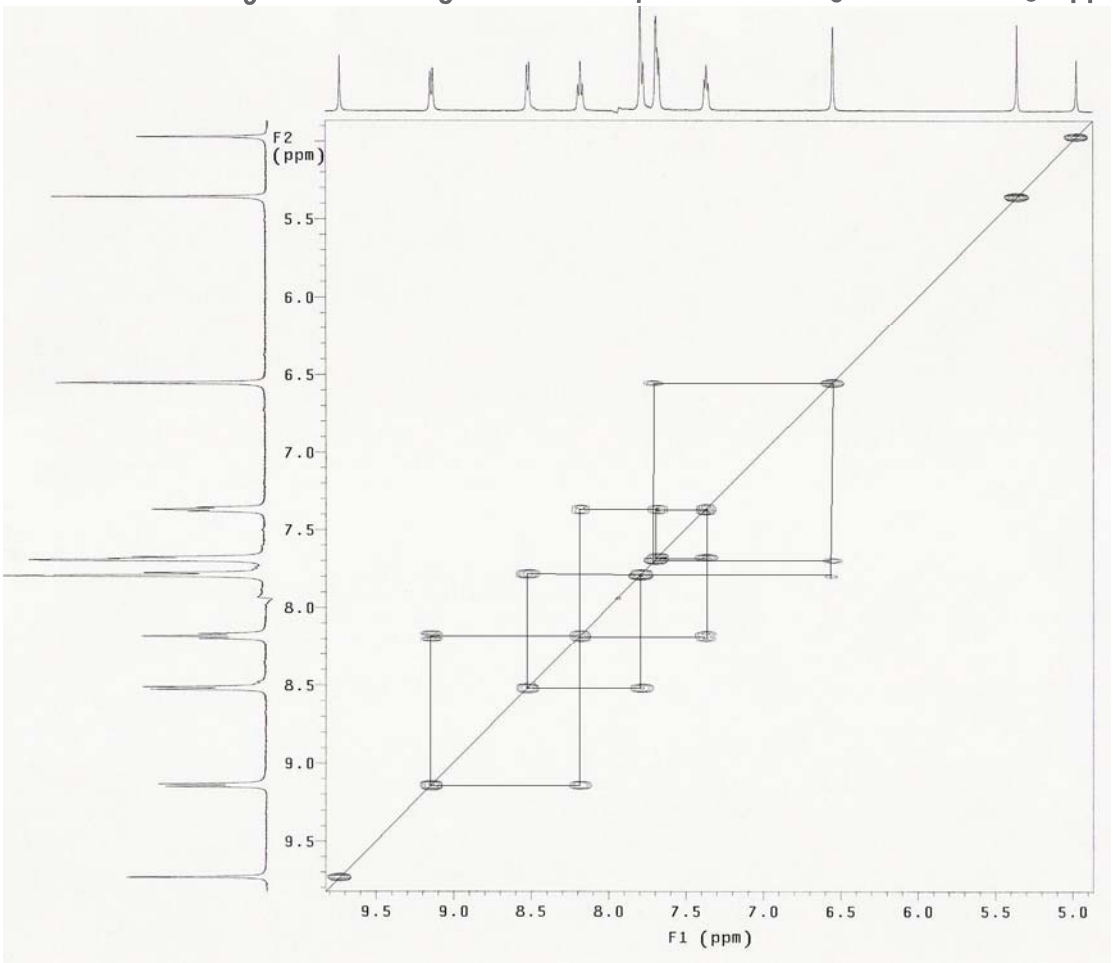


Figure 4.13. (A) ^{1}H NMR spectrum of complex **4.6**. (B) ^{1}H-^{1}H COSY of $[Fe(pzt)_2](PF_6)_2$ in acetone-d_6 solution.

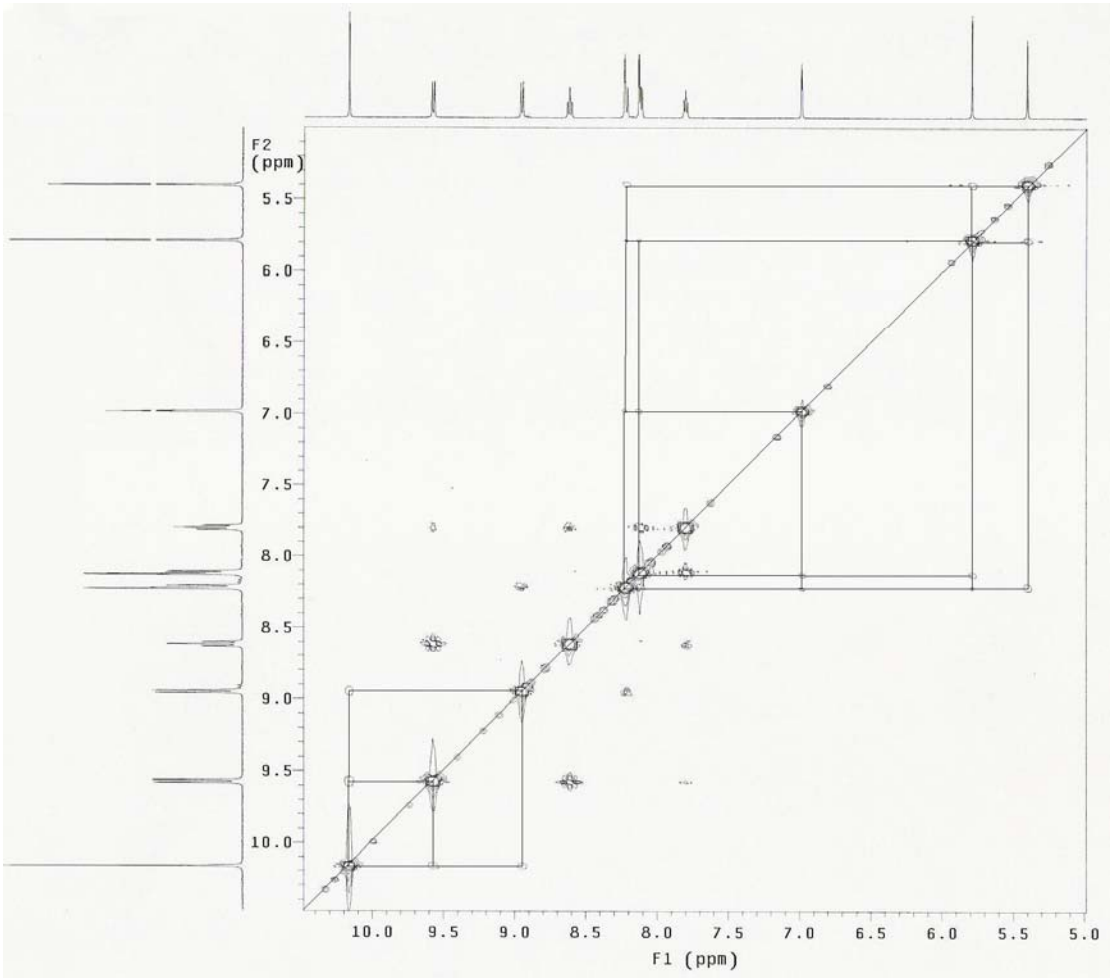


Figure 4.14. ^{1}H-^{1}H NOESY of $[Fe(pzt)_2](PF_6)_2$, **4.6**, in acetone-d_6 solution

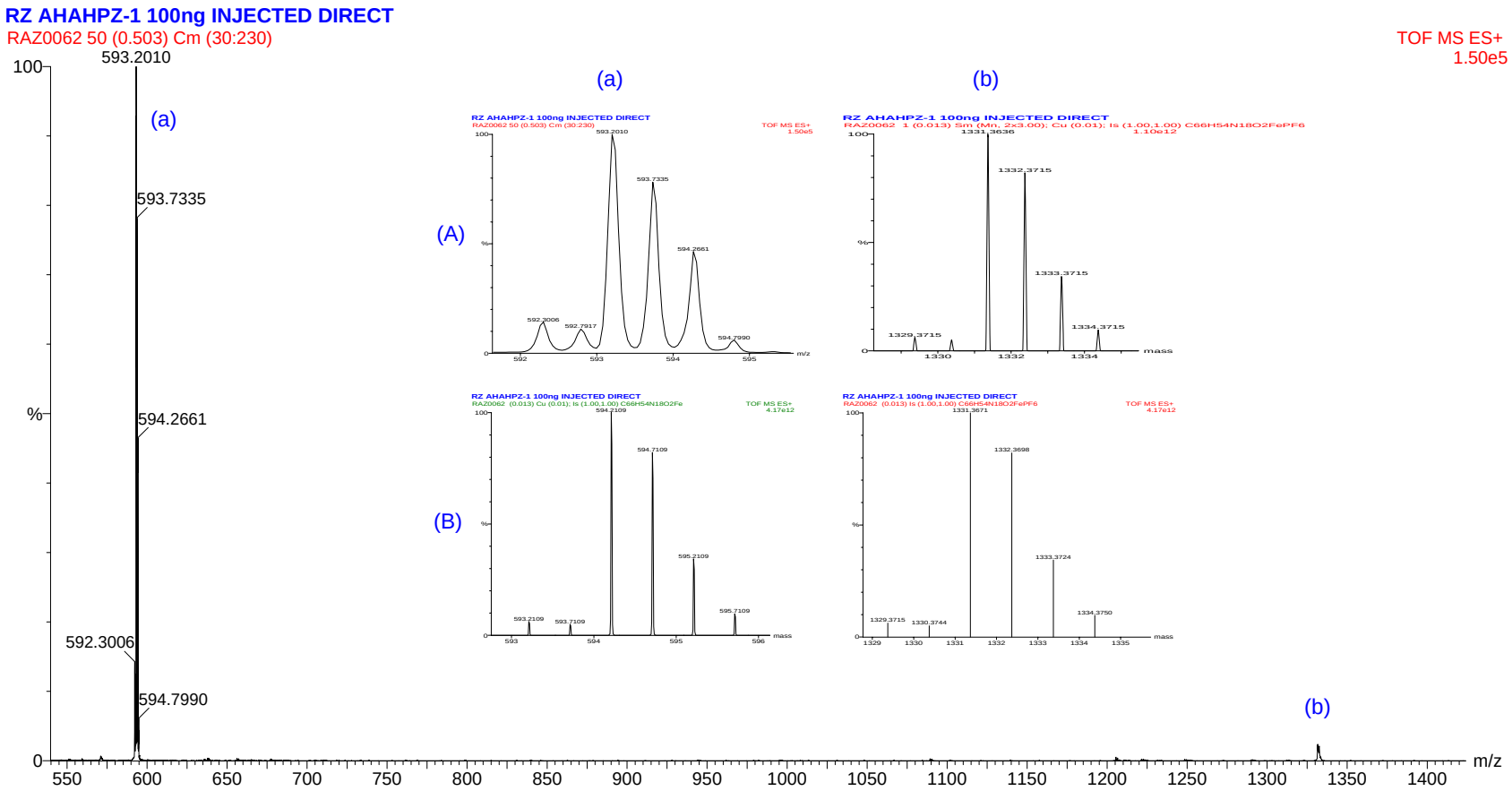


Figure 4.15. ESI-MS of $[Fe(pzt)_2](PF_6)2$ complex, **4.6**, in CH_3CN. (A) High resolution scans of the two major signals (a) and (b); (B) Calculated isotope distribution patterns for (a) $[Fe(pzt)_2]^{2+}$ and (b) $[Fe(pzt)_2+(PF_6)]^{+}$ at *m/z* 593.2 and 1331.6, respectively.

Once again, a good refinement of an X-ray crystallographic structural model eluded us, but the result was consistent with the proposed coordination mode. The relative ease of synthesis and purification for this complex made it the preferred choice for the attempts to prepare heteronuclear systems that are described later in this chapter.

4.3.3. Copper Complex of pzt Ligand

Complex **4.7**, $[Cu(pzt)(NO_3)_2]$, was synthesised by layering of a $Cu(NO_3)_2$ solution in MeOH onto a CH_2Cl_2 solution of the pzt ligand. After two weeks, green-blue crystals of the complex were formed. The complex was characterised both in the solid state (X-ray crystallography and infrared spectroscopy) and in solution (ESI-MS).

4.3.4. Zinc and Cadmium Complexes

Zinc complexes of the pzt ligand can also be formed. Mixing different ratios of the pzt ligand and $ZnCl_2$, in $MeOH/CH_2Cl_2$, produced the same white powder, regardless of whether the metal or the ligand is in excess. The elemental analysis of this powder is consistent with the formulation $[Zn(pzt)Cl_2]$ for complex **4.8** and the NMR data obtained for the complex are consistent with there being only one terpyridine fragment coordinated to the zinc ion (there is no up-field shift associated with the $H_{6,6''}$ and $H_{5,5''}$ protons).[384]

Recrystallisation of the powder by slow evaporation of a CH_3CN/CH_2Cl_2 solution gave X-ray quality crystals. Identical crystalline material can be obtained

directly if the complexation reaction is conducted in mixed CH_3CN/CH_2Cl_2 solvent that is then allowed to evaporate. We note, however, that ESI-MS results for both the powder or crystalline material dissolved in dmso and the CH_3CN/CH_2Cl_2 reaction mixture reveal large signals that can be assigned to a $[Zn(pzt)_2]^{2+}$ complex and free ligand, and only very small signals that can be assigned to complexes containing only a single ligand. The solution state data obtained from NMR experiments (in particular the lack of up-field shifts that would be expected for the $H_{6,6''}$ and $H_{5,5''}$ protons if they were affected by the magnetic field associated with a second coordinated terpyridine ligand) is entirely consistent with the $[Zn(pzt)Cl_2]$ structure derived from the crystallographic study and the composition of the complex derived from elemental analysis. This therefore represents something of a warning regarding the significance that should be attached to ESI-MS data when they are unsupported by other evidence.

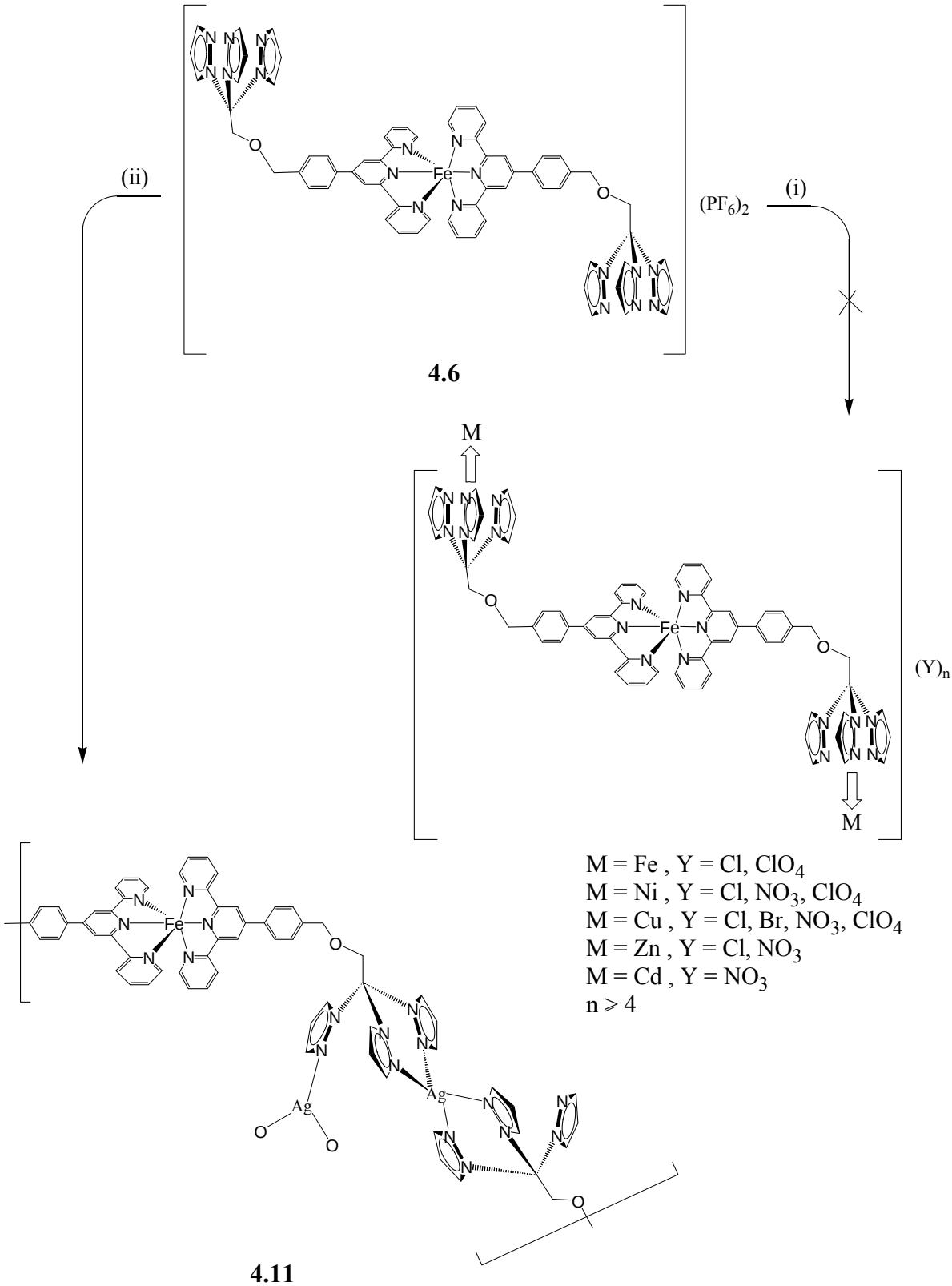
A cadmium complex of the pzt ligand can be synthesized by following a similar approach that used for preparation of the zinc complex (Scheme 4.2). $Cd(NO_3)_2$ in CH_3CN was reacted with the pzt ligand in CH_2Cl_2 to obtain a clear solution. The solution was taken to dryness and the crude product recrystallised from MeOH-diethyl ether. Needle-like crystals of a complex were produced by slow evaporation of CH_3CN/CH_2Cl_2 solution of the reaction mixture, but they were not suitable for X-ray crystallographic analysis. The ESI-MS exhibits peaks at *m/z* 1305.3 and 622.3, that can be assigned to the $[Cd(pzt)_2(NO_3)]^+$ and $[Cd(pzt)_2]^{2+}$ cations, respectively. A small peak at *m/z* 739.2 corresponding to $[Cd(pzt)]^+$ was also observed. However, ^1H NMR spectroscopy and elemental analysis are entirely consistent with the bulk material having only one pzt ligand per cadmium ion, and a formula of $[Cd(pzt)(NO_3)_2]$. The cadmium chemistry appears to be rather similar to that of zinc that is described above.

4.3.5. Heteronuclear Complexes

During our studies of the coordination chemistry of the pzt ligand, we could find no evidence for coordination of the tris(pyrazolyl) portion of the ligand to any metal ion other than silver(I). It is not surprising, therefore, that our only positive results from attempted syntheses of heteronuclear systems proved to be those in which silver(I) was bound to the tris(pyrazolyl) site.

The observed preference for all metal ions studied, including silver(I), to bind to the terpyridyl binding site led us to concentrate on heteronuclear systems where metal ions are already coordinated in that site. That is, the terpyridyl complexation must be in place before binding to the pyrazolyl portion of the molecule can be attempted, at least for labile metal ions. The iron(II) complex, $[Fe(pzt)_2](PF_6)_2$, **4.6**, is readily prepared and isolated, and the complex is both diamagnetic and sufficiently inert to allow study of complexation to the tris(pyrazolyl) binding site. This complex was therefore at the centre of these studies.

Reactions of **4.6** with $AgNO_3$, $AgClO_4$, and $AgBF_4$ were examined (Scheme 4.3).



Scheme 4.3. Reactions of complex **4.6** with different transition metal ions. (i) MX_2 (M = Fe, Ni, Cu, Zn, Cd; X = Cl, NO_3) in appropriate solvents. (ii) $AgClO_4$, CH_2Cl_2/CH_3CN, under Ar in dark, r.t.

When **4.6** was allowed to react with $AgNO_3$, a purple microcrystalline compound was precipitated upon slow evaporation of the reaction mixture. ^1H NMR data are consistent with coordination at the pyrazolyl site (Figure 4.16), and ESI-MS of this material indicated that polymeric material was present, in addition to signals that can be assigned to simpler silver containing species (with good matches to predicted isotope patterns). Representative mass spectra are shown in Figure 4.17 and Figure 4.18. Since the colour of the complex is unchanged, we infer that the bis(terpyridyl) coordination environment around the iron is unchanged, and that coordination of silver(I) is occurring at the pyrazolyl site (see Table 4.1). Needle-like crystals were obtained within a few days after layering a CH_3CN solution of $AgNO_3$ onto a CH_2Cl_2 solution of complex **4.6**. Unfortunately the crystals were not suitable for X-ray crystallography.

Similar results were observed when $AgBF_4$ or $AgClO_4$ were used instead of $AgNO_3$ in other reactions with **4.6**. Small needle-like crystals were again obtained, this time by slow evaporation of the reaction mixture of $AgClO_4$ with **4.6**. X-ray diffraction of a very small single crystal revealed that a 1D zigzag polymer containing iron and silver ions was formed (Scheme 4.3), but the marginal quality of the crystals meant that while a structural solution could be found, the refinement was unsatisfactory ($R_1 \approx 18\%$). The tentative structure, shown in Scheme 4.3, is that of a coordination polymer, with iron bound in the terpyridyl sites of two ligands, one silver ion linking the iron complexes through coordination to two pyrazolyl groups, and a second silver ion coordinated to the remaining pyrazolyl group of one ligand. We expect, based on the chemistry described above, that the polymeric species will be rather labile in solution, and this structure should probably only be interpreted as an indicator of the kinds of structure that are present.

Addition of silver salts to the pzt complexes of ruthenium(II), resulted in some changes in the chemical shifts of the pyrazolyl ring protons in the ^{1}H NMR spectra. ESI-MS results show that polymeric species are again formed, and this, together with the NMR results, allows us to infer that silver ions are coordinating to the tris(pyrazolyl) site of the ligand.

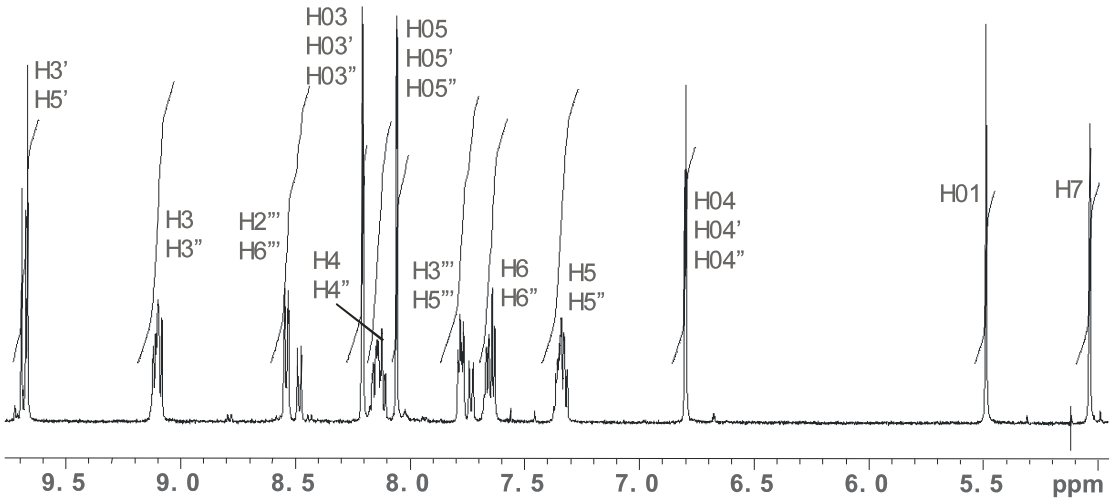


Figure 4.16. ^{1}H NMR spectrum of the polymer obtained from the reaction of **4.6** with $AgNO_3$.

Compound / Proton	**1.71**[*]	**1.71**[†]	**4.2**[†]	**4.4**[‡]	**4.5**[‡]	**4.6**[‡]	**4.6**[†]	**4.8**[†]	**4.9**[†]	**4.11**[†]	**A**[*]	**A**[†]	**B**[†]	**C**[†]	**D**[†]	**E**[‡]	**F**[‡]
6/6"	8.67	8.77	8.50	7.93	7.94	7.35	7.68	8.95	8.85	7.66	8.60	8.71	7.16[§]	7.90	8.8 [§]	8.94 [§]	7.90
5/5"	7.36	7.63[§]	7.59	7.47	7.47	7.28	7.37	7.95	7.98	7.34	7.22	7.50	7.16[§]	7.45	7.90	7.90	7.48
4/4"	7.89	8.14	8.17	8.23	8.22	8.12	8.18	8.39	8.45	8.15	7.75	7.99	7.95	8.23	8.32	8.38	8.21
3/3"	8.73	8.86	8.67	9.18	9.18	9.14	9.15	8.97	9.11	9.10	8.51	8.59	8.82	8.95	8.8 [§]	8.94 [§]	8.87
3'/5'	8.72	8.80	8.73	9.55	9.55	9.75	9.74	9.09	9.15	9.67	8.22	8.25	9.03	8.83	8.59	8.58	8.81
2'''/6'''	7.86	7.99	8.20	8.41	8.41	8.60	8.51	8.27	8.31	8.63							
3'''/5'''	7.30	7.48	7.51	7.74	7.74	7.70	7.78	7.48	7.55	7.78							
7	4.61	4.74	4.79	4.95	4.95	4.91	4.97	4.78	4.80	5.04							
01	5.20	5.17	5.19	5.35	5.34	5.23	5.36	5.19	5.17	5.49							
03/03'/03"	7.68	7.79	7.81	7.79	7.79	7.83	7.80	7.81	7.79	8.21							
04/04'/04"	6.36	6.53	6.55	6.55	6.55	6.57	6.55	6.54	6.53	6.80							
05/05'/05"	7.46	7.63[§]	7.65	7.69	7.69	7.67	7.70	7.65	7.63	8.06							

Table 4.1. Chemical shifts (δ) (ppm) for pzt ligand, its complexes, and their comparison with the literature values.[384]

A, 4'-methylthio-2,2':6',2"-terpyridyl[384]; **B**, Fe**A**$_2$(ClO$_4$)$_2$[384]; **C**, Zn**A**$_2$(ClO$_4$)$_2$[384]; **D**, Zn**A**Cl$_2$[384]; **E**, Zn**A**(NO$_3$)$_2$[384]; **F**, Zn**A**$_2$(NO$_3$)$_2$[384].

[*] $CDCl_3$
[†] dmso-d_6
[‡] acetone-d_6
[§] Signals overlap. Chemical shift values should be treated as ± 0.05.

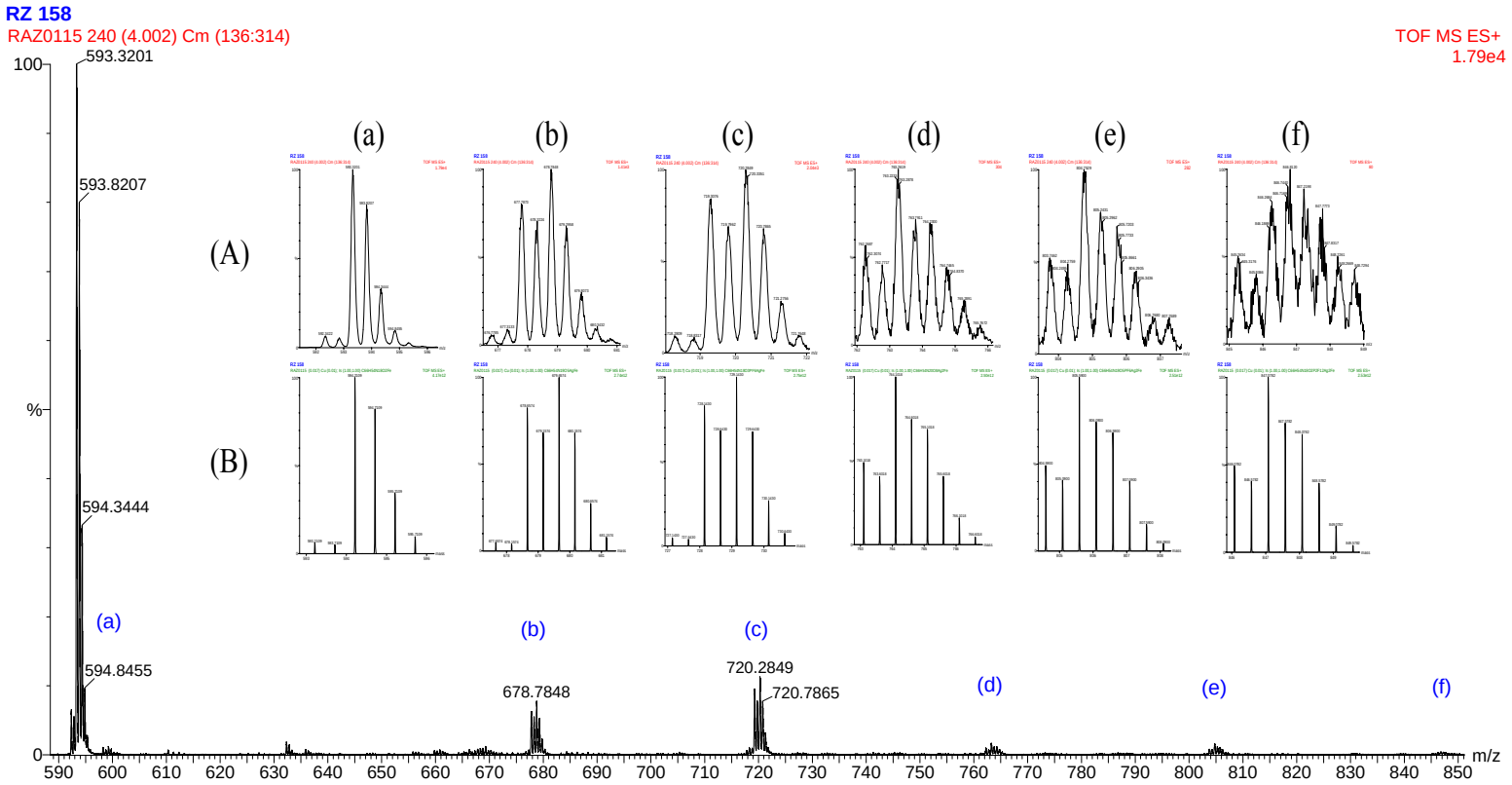


Figure 4.17. ESI-MS of material obtained from the reaction of **4.6** with $AgNO_3$ in acetone. (A) High resolution scans of the six major signals (a)-(f); (B) Calculated isotope distribution patterns for (a) $[Fe(pzt)_2]^{2+}$, (b) $[Fe(pzt)_2AgNO_3]^{2+}$, (c) $[Fe(pzt)_2AgPF_6]^{2+}$, (d) $[Fe(pzt)_2Ag_2(NO_3)_2]^{2+}$, (e) $[Fe(pzt)_2Ag_2(NO_3)(PF_6)]^{2+}$, (f) $[Fe(pzt)_2Ag_2(PF_6)_2]^{2+}$at *m/z* 593.3, 678.8, 720.3, 763.3, 804.8, and 846.8, respectively.

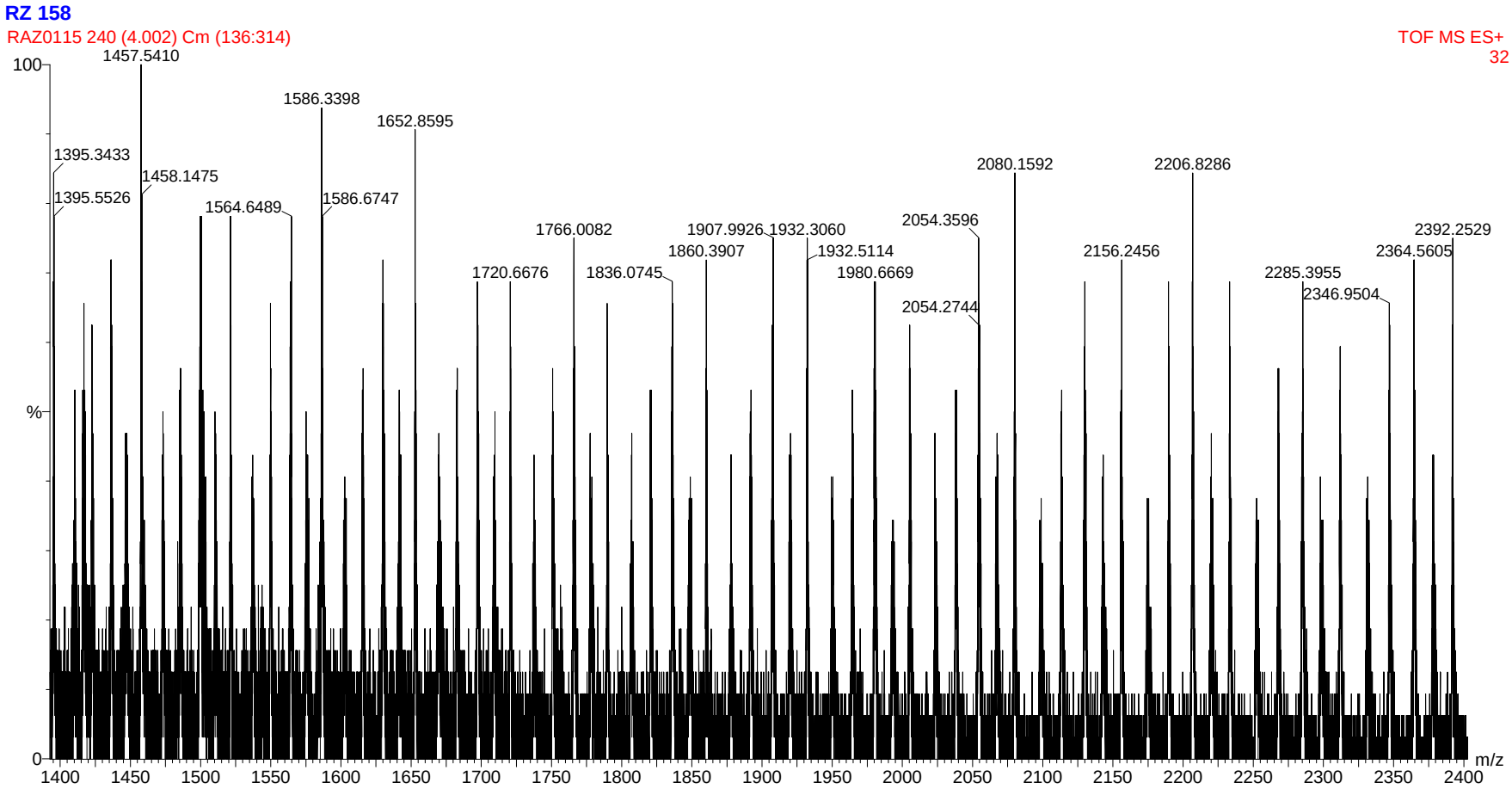


Figure 4.18. Expanded region (*m/z* 1400 to 2400) of ESI-MS data for the material formed in the reaction of **4.6** with $AgNO_3$ in acetone.

In contrast, addition of silver salts to the cadmium(II), and zinc(II) complexes of pzt ligand leads to formation of the silver complex **4.2**, based on NMR and ESI-MS results. There was no evidence of any coordination to the tris(pyrazolyl) site when [Zn(pzt)Cl_2], **4.8**, or [Cd(pzt)$(NO_3)_2$], **4.9**, was treated with silver(I) salts, as chemical shifts of the pyrazolyl rings protons in the ^{1}H NMR spectra remain almost unchanged.

^{1}H NMR titrations of the zinc and cadmium complexes, **4.8** and **4.9**, with $AgBF_4$, in dmso-d_6 solution, were also examined in different experiments (Figure 4.19).

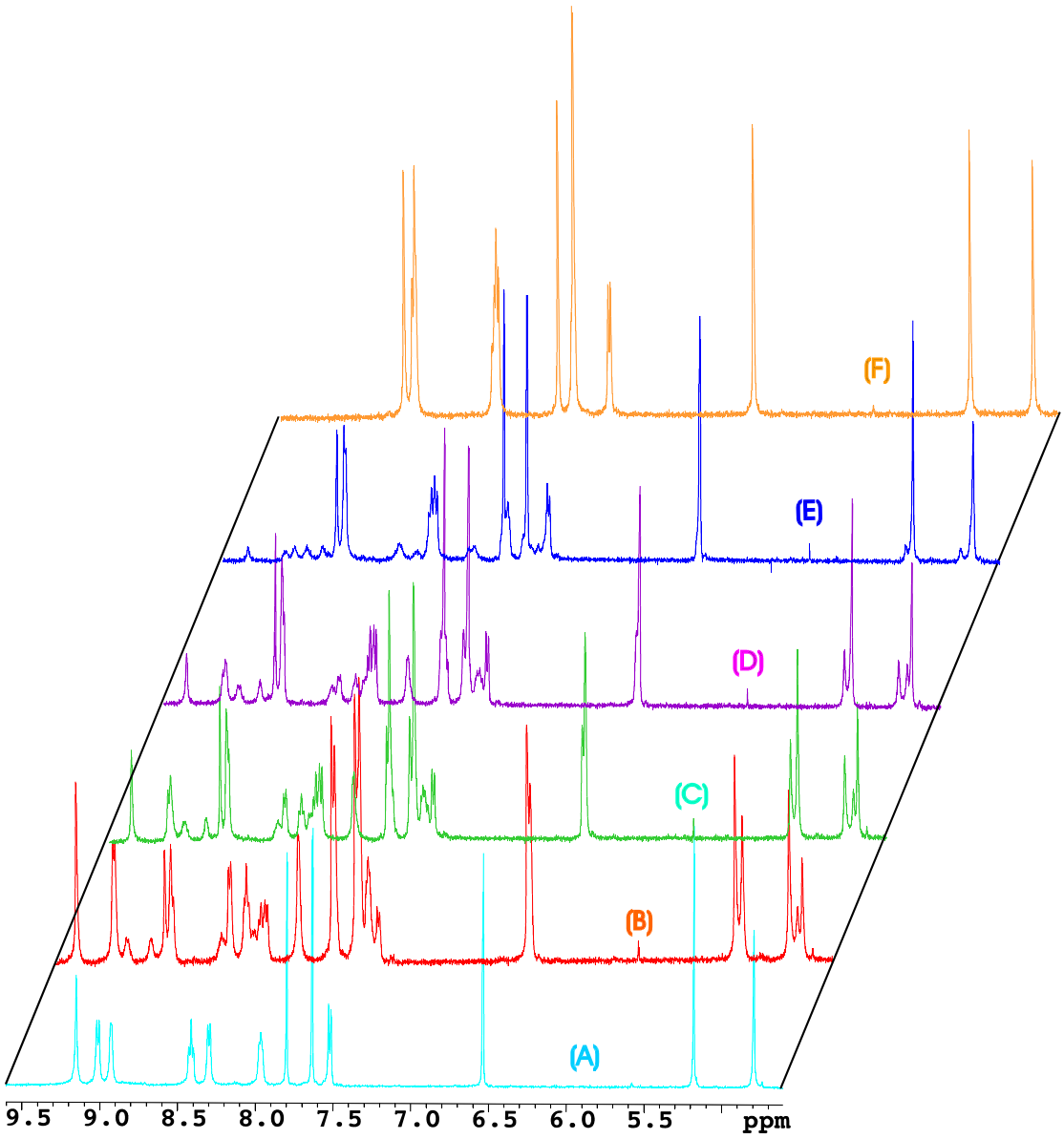


Figure 4.19. ^{1}NMR titration of [Zn(pzt)Cl_2] in dmso-d_6 with $AgBF_4$ solution in dmso-d_6. **(A)** [Zn(pzt)Cl_2] in dmso-d_6; **(B)** [Zn(pzt)Cl_2] + $AgBF_4$ in dmso-d_6 (1:3); **(C)** [Zn(pzt)Cl_2] + $AgBF_4$ in dmso-d_6 (1:5); **(D)** [Zn(pzt)Cl_2] + $AgBF_4$ in dmso-d_6 (1:7); **(E)** [Zn(pzt)Cl_2] + $AgBF_4$ in dmso-d_6 (1:11); **(F)** [Zn(pzt)Cl_2] + $AgBF_4$ in dmso-d_6 (1:20).

In the case of compound [Cd(pzt)$(NO_3)_2$], **4.9**, only one set of signals were observed throughout the experiment. In the case of compound [Zn(pzt)Cl_2], **4.8**, two sets of signals, corresponding to the compounds [Zn(pzt)Cl_2], **4.8,** and

$[Ag_2(pzt)_2](BF_4)_2$, **4.2,** were observed, except when a large excess of silver salt was present in the solution. In the latter case, only one set of signals, corresponding to the silver complex $[Ag_2(pzt)_2](BF_4)_2$, **4.2,** was observed. These results can be attributed to the ligand exchange process in the case of zinc(II) complex being slow on the NMR time scale compared to those for the labile cadmium(II) and silver(I) complexes, which are fast on the NMR time scale. Subsequent ^1H NMR experiments in which the pzt ligand was titrated with $ZnCl_2$ were performed (Figure 4.20) and these showed two sets of signals, corresponding to the free ligand and $[Zn(pzt)Cl_2]$ complex, **4.8**, until sufficient zinc has been added to convert all of the ligand to its zinc complex, **4.8**.

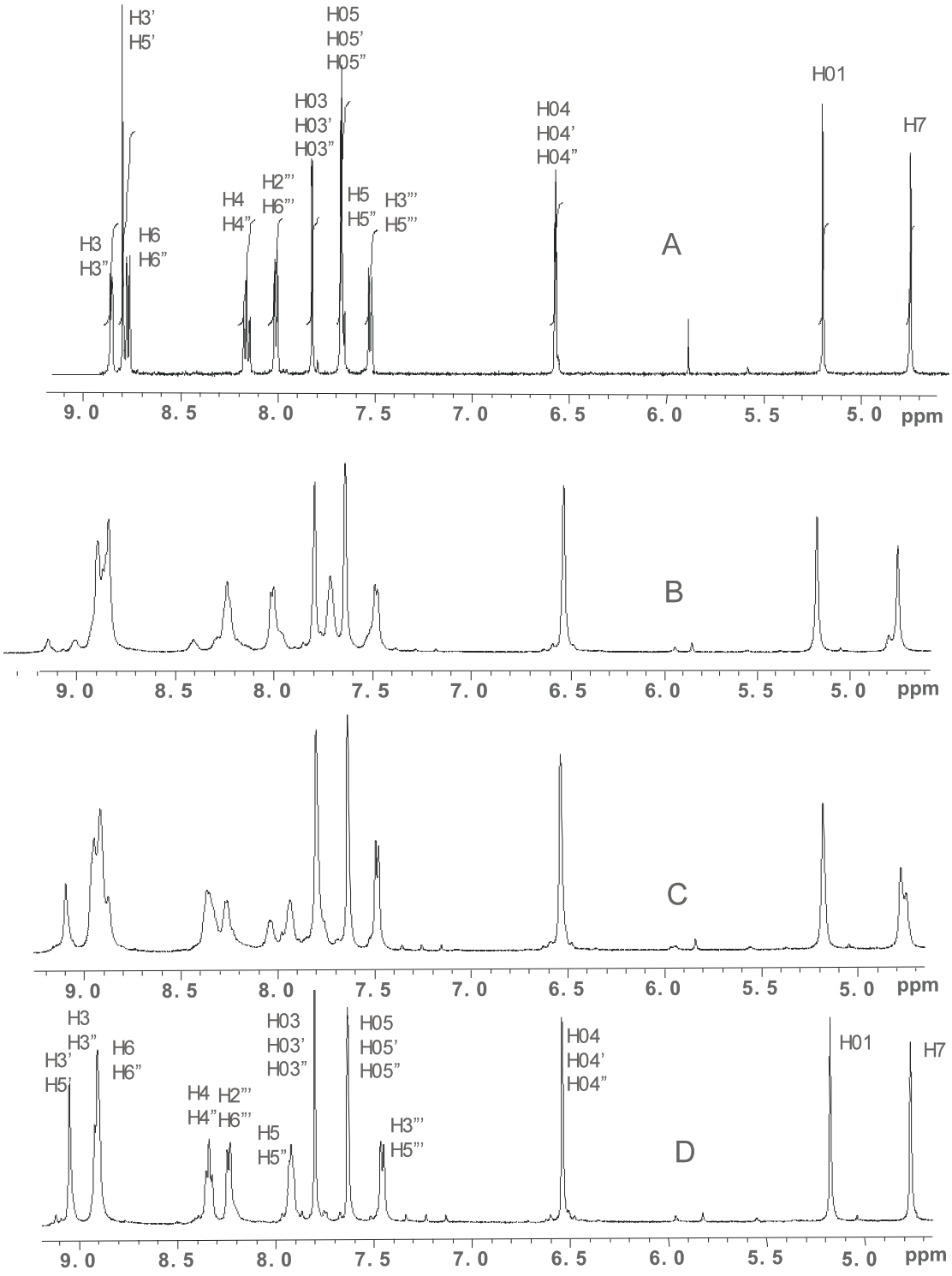


Figure 4.20. ^{1}H NMR titration of pzt ligand in dmso-d_6 with $ZnCl_2$ solution in dmso-d_6: **A** ^{1}NMR of pzt ligand in dmso-d_6. **B** ^{1}NMR of pzt + $ZnCl_2$ in dmso-d_6 (L:M)(1:0.1). **C** ^{1}NMR of pzt + $ZnCl_2$ in dmso-d_6 (L:M)(1:0.5). **D** ^{1}NMR of **3.9** in dmso-d_6 (L:M)(1:1)

As also shown in Figure 4.21, significant changes in the ^{1}HNMR chemical shifts corresponding to the protons H_3, H_3'', H_4, H_4'', H_5, H_5'', H_6, H_6'', H_3', and H_5' were observed. These observations indicated coordination of zinc ion at the terpyridine site of the ligand pzt.

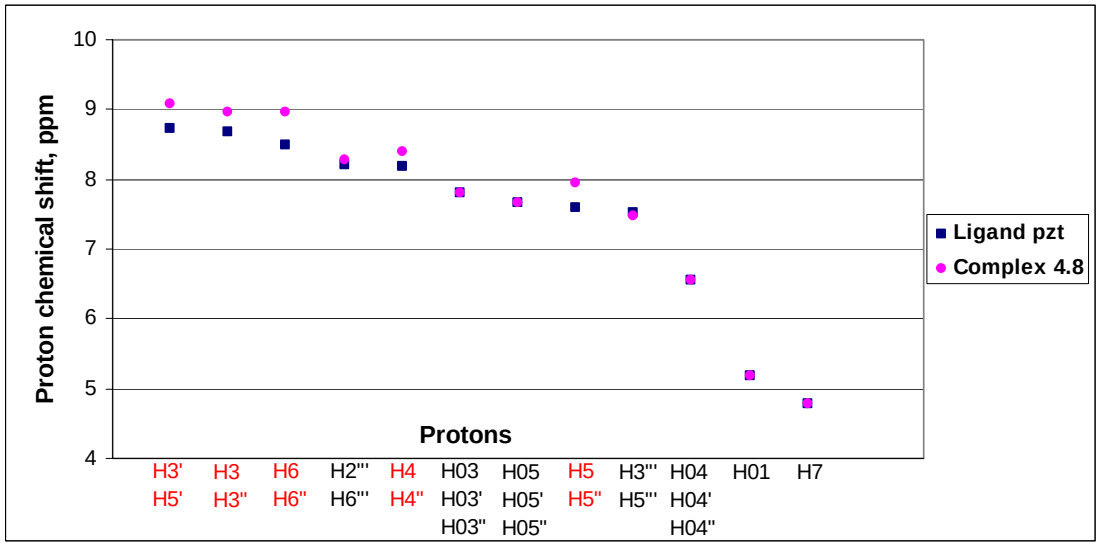


Figure 4.21. Comparison of the ^{1}H NMR chemical shifts of the ligand pzt and its zinc complex **4.8** solutions in dmso-d_6.

ESI-MS results for both sets of silver titrations in dmso/MeOH solutions reveal signals that can be assigned to a $[M(pzt)_2]^{2+}$ complex (where M = Zn or Cd), $[Ag_2(pzt)_2]^{2+}$ complex, and free ligand. The ratios of the peaks are proportional to the concentrations of Zn/Cd and Ag salts that are present in the solutions. These results are entirely consistent with those seen in the NMR studies.

There was no evidence of any coordination to the tris(pyrazolyl) site when $[Fe(pzt)_2](PF_6)_2$ complex, **4.6**, was treated with $CuBr_2$, $CuCl_2$, $Cu(NO_3)_2$, $Cu(ClO_4)_2$, $NiCl_2$, $Ni(NO_3)_2$, $Ni(ClO_4)_2$, $FeCl_2$, $Fe(ClO_4)_2$, $ZnCl_2$, $Zn(NO_3)_2$, $Zn(SO_4)_2$, $Cd(NO_3)_2$, $CoCl_2$, $Na_3[Co(NO_2)_6]$, or $Co(NO_3)_2$ under a variety of conditions. In some cases,

precipitates were formed, but these proved to be $[Fe(pzt)_2]^{2+}$ complexes with different anions. There was no evidence for any polynuclear material in the ESI-MS of the reaction mixtures, and there were no changes in the ^1H NMR spectra of the $[Fe(pzt)_2]^{2+}$ complex when those metal ions that are diamagnetic were added to the solution.

4.4. X-Ray Crystallographic Studies of Some Metal Complexes of the 4'-(4-(2,2,2-Tris(1*H*-pyrazol-1-yl)ethoxymethyl)phenyl)-2,2':6',2''-terpyridine (pzt) Ligand

4.4.1. Crystal Structure of the pzt Ligand, 1.71

Crystals suitable for X-ray diffraction were obtained by layering a dichloromethane solution of the ligand with hexanes. The ligand was crystallised in orthorhombic *Pbca* space group (R_1 = 0.06). As shown in Figure 4.22, the three pyridyl rings are approximately coplanar, making an interannular angle with the central pyridine ring of 11.5º, with a *transoid* arrangement about each interannular C-C bond. This is in accord with the *transoid* configuration observed in the crystal structures of 2,2'-bipyridine[447-449] , 2,2':6',2'':6'',2'''-quaterpyridine[233] and 4'-phenyl-2,2':6',2''-terpyridine[187]. The C-C and C-N distances within the phenyl and the pyridine rings are normal (average 1.307 and 1.351 Å, respectively). The interannular C-C bonds also closely resemble those seen in 2,2'-bipyridine[447-449], 2,2':6',2'':6'',2'''-quaterpyridine[233], and 4'-phenyl-2,2':6',2''-terpyridine[187] with lengths of 1.498 Å.

The phenyl ring is not coplanar with the terpyridyl fragment, but is twisted about the interannular bond such that the plane of the phenyl ring makes an angle of 24.5º with the plane of the central pyridine ring. This represents a compromise between a coplanar arrangement in which π-conjugation and non-bonded H-H contacts between the *ortho* protons of the phenyl ring and H2 H4 are maximized. This angle is larger than that of 4'-phenyl-2,2':6',2"-terpyridine structure (10.9º). One of the pyrazole rings of the pyrazolyl site is approximately in the plane of C7-O-C01 linker with a slight twist of 10.6º. The angles between the remaining pyrazole rings and the plane of the etheric chain linker are 76.3º and 82.1º, respectively. The free nitrogen atoms of the pyrazole rings are all pointed out from each other in such a way that the interactions between the hydrogen atoms of the pyrazolyl rings and the free nitrogen atoms at the adjacent rings are small.

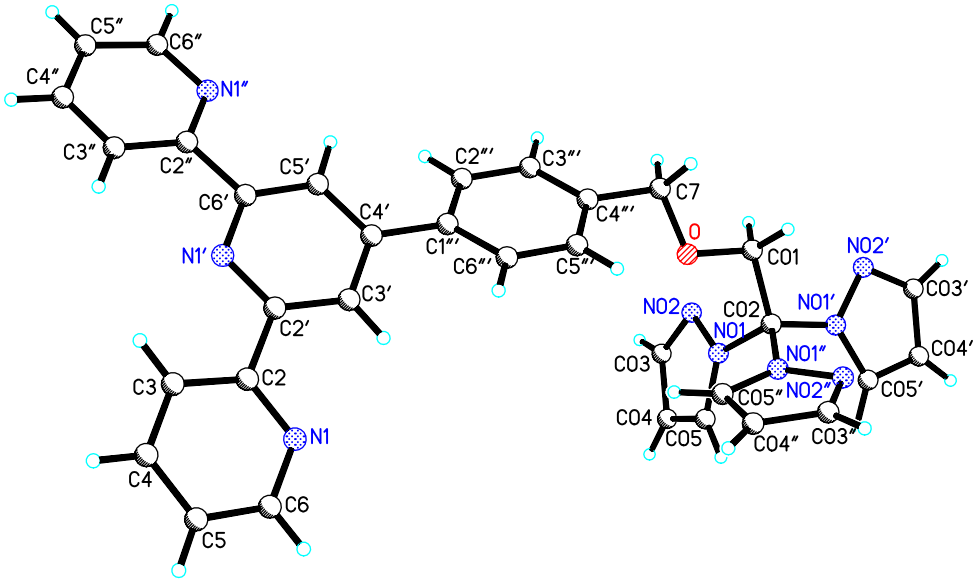


Figure 4.22. Molecular structure of ligand pzt, **1.71**. Selected bond lengths [Å] and angles [°]: N(1)-C(6) 1.340(4), N(1)-C(2) 1.358(4), N(1')-C(6') 1.348(4), N(1')-C(2') 1.361(4), N(1")-C(2") 1.338(4), N(1")-C(6") 1.358(4), N(01)-N(02)

1.369(4), N(01)-C(05) 1.392(4), N(01)-C(02) 1.490(4), N(02)-C(03) 1.349(5), N(01')-N(02') 1.354(4), N(01')-C(05') 1.370(4), N(01')-C(02) 1.447(4), N(01")-C(05") 1.369(4), N(01")-N(02")1.379(4), N(01")-C(02) 1.457(4), N(02")-C(03") 1.320(4), O-C(01) 1.414(4), O-C(7) 1.415(4), C(01)-O-C(7) 113.0(2), O-C(7)-C(4"') 107.6(3), O-C(01)-C(02) 105.5(3).

As shown in Figure 4.23, the solid state structure is stabilised by hydrogen bonding interactions between pyrazolyl groups of one molecule with pyridine and pyrazolyl groups from adjacent molecules. There is no obvious π-π stacking interaction between the molecules.

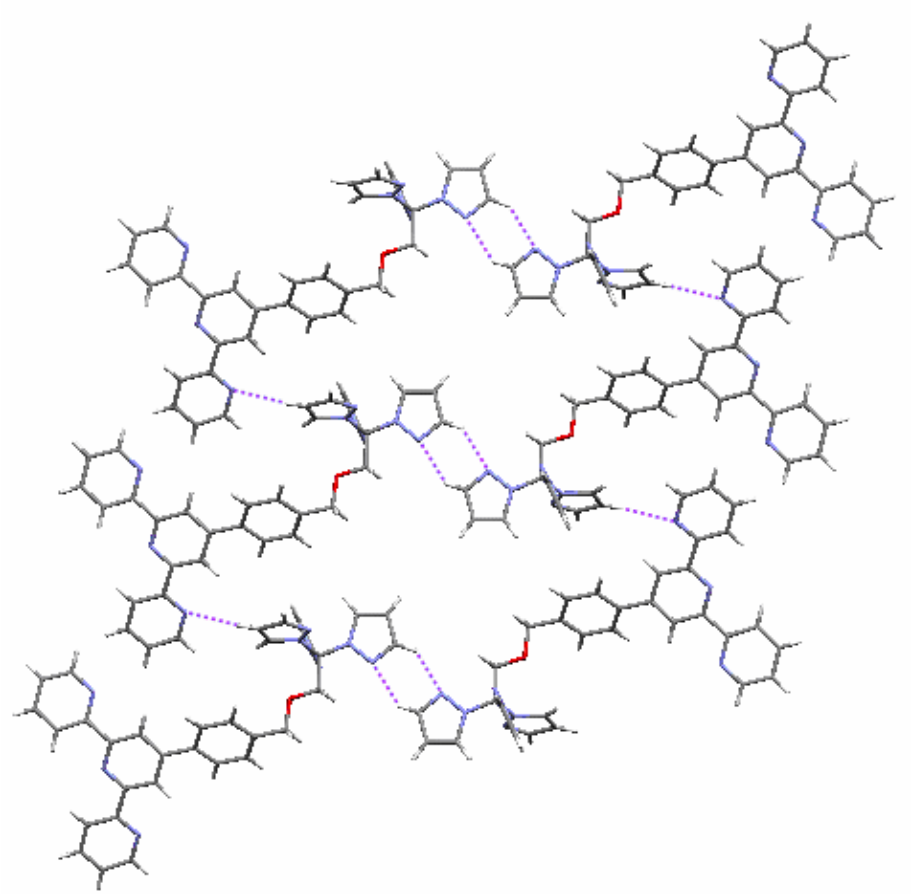


Figure 4.23. Hydrogen bonding network within the lattice of the structure ligand pzt, **1.71**. Hydrogen bonds are shown using the purple dotted lines.

4.4.2. Crystal Structure of $[Ag_2(pzt)_2](BF_4)_2$, 4.2

Colourless single crystals suitable for X-ray crystallography diffraction were obtained by slow evaporation of dmso-d_6 solution of the complex at r.t. The complex was crystallised in monoclinic $P2_1/c$ space group. As shown in Figure 4.24, each Ag ion is coordinated to tridentate terpyridine site of the ligand **1.71** in the complex. The fourth coordination site of each Ag ion is also occupied by one pyrazole ring of non-tripodal pyrazolyl site of the ligand **1.71**. The terpyridine site acts as a planar tridentate binding moiety while the pyrazolyl site behaves as a monodentate system to coordinate to silver ion to form a [2 + 2] complex. The two remaining pyrazolyl rings remain non-coordinated. There are π-π stacking interactions (face-face, centroid-centroid) between the adjacent dimers by which the structure is stabilised (Figure 4.25). The separation between a central pyridine ring of a dimer to the phenyl ring of the adjacent dimer is 3.6 Å. The distance between the coplanar phenyl terpyridine planes in a dimer is about 3.5 Å.

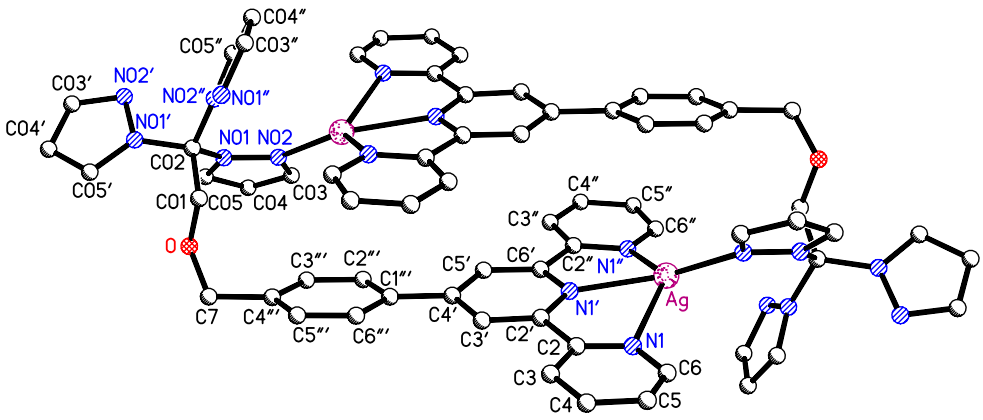


Figure 4.24. Crystal structure of a [2 + 2] silver complex, **4.2,** with numbering scheme adopted. Hydrogen atoms and disordered BF_4^- anions are omitted for clarity. Selected bond lengths [Å] and angles [°]:Ag-N(02)#1 2.236(4), Ag-N(1')

2.329(3), Ag-N(1") 2.372(4), Ag-N(1) 2.586(4), N(02)-Ag#1 2.236(4), N(02)#1-Ag-N(1') 144.67(14), N(02)#1-Ag-N(1") 137.33(13), N(1')-Ag-N(1") 70.27(13), N(02)#1-Ag-N(1) 89.31(14), N(1')-Ag-N(1) 66.16(14), N(1")-Ag-N(1) 133.25(14), C(2)-N(1)-Ag 114.6(3), C(6)-N(1)-Ag 126.3(4), C(6')-N(1')-Ag 118.4(3), C(6")-N(1")-Ag 125.0(3), C(2")-N(1")-Ag 116.1(3), C(03)-N(02)-Ag#1 122.5(3), N(01)-N(02)-Ag#1 133.3(3). [Symmetry code: (#1) 1-x, -y, 2-z].

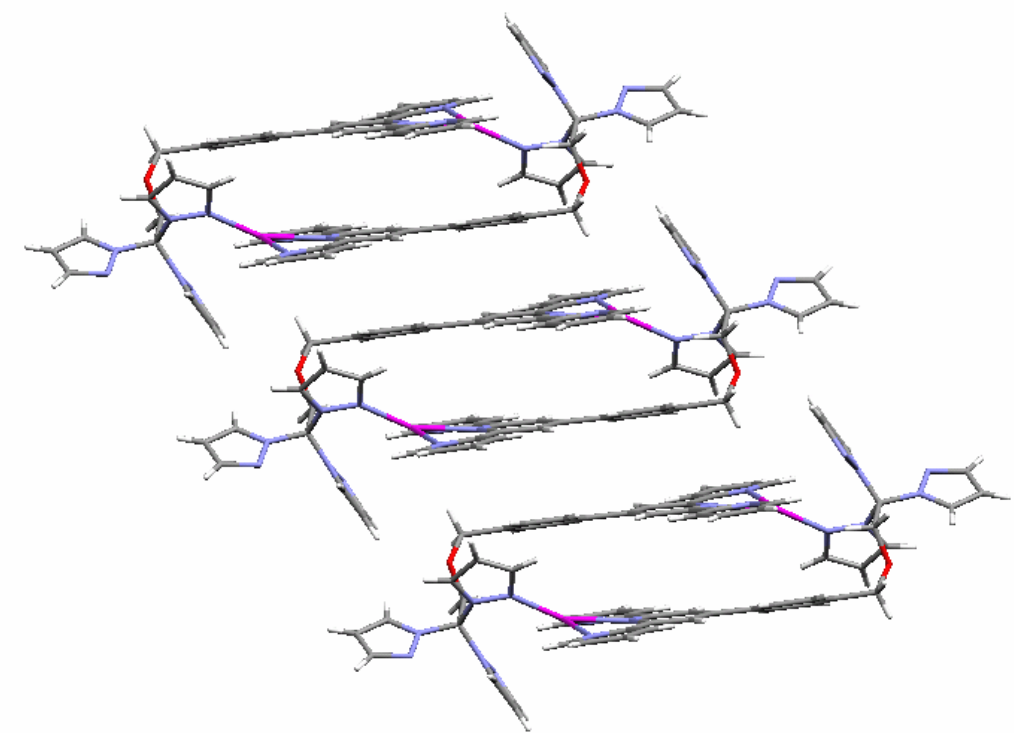


Figure 4.25. π-π stacking interactions between the dimers of the silver complex **4.2**. The distance between the planes of the adjacent molecules is about 3.6 Å.

4.4.3. Crystal Structure of $[Cu(pzt)(ONO_2)_2].CH_3CN$, 4.7

When a solution of $Cu(NO_3)_2.3H_2O$ in CH_3CN was layered onto a solution of **1.71** in DCM, green crystals suitable for X-ray diffraction crystallography were

obtained over a week in the refrigerator followed by slow evaporation. The complex crystallised in $P\bar{1}$ space group. The crystal structure revealed that the Cu ion was coordinated to the tridentate terpyridine site while the pyrazolyl site remained uncoordinated (Figure 4.26). The coordination sphere around the copper ion is completed by two monodentate nitrate anions. The geometry around the metal centre lies between that of a square pyramid (with the three terpyridine nitrogen donors of the pzt ligand and the oxygen donor atom of one nitrate ligand forming the equatorial plane and the other nitrate ligand occupying the apical position) and a trigonal bipyramid (where the two nitrate ligands and the central nitrogen donor of the terpyridine fragment form the trigonal plane). This is not an unusual situation, as there are examples of mono(terpyridyl)copper(II) complexes in the literature in which the geometry of the copper(II) ions are described as either trigonal bipyramidal[220,391] or square pyramidal[337,450-457].

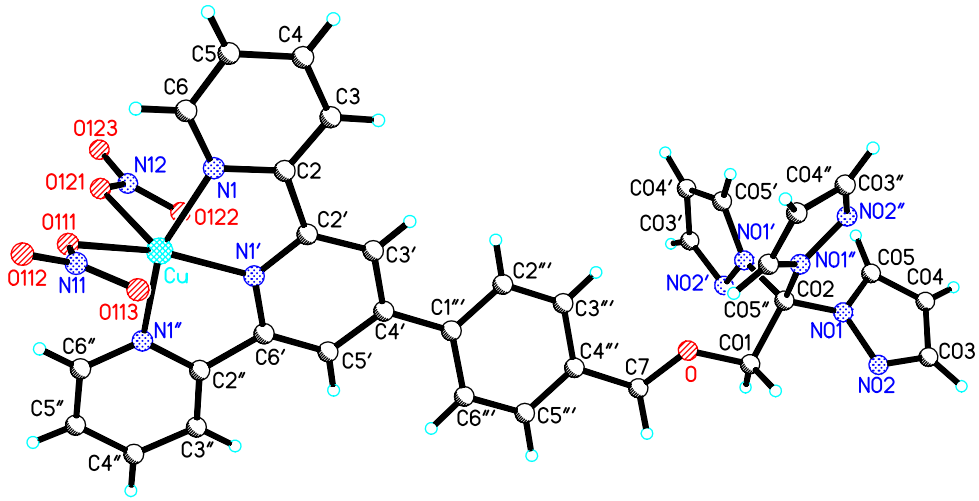


Figure 4.26. Molecular structure of the neutral Cu complex **4.7** with a disordered CH_3CN molecule omitted for clarity. Selected bond lengths [Å] and angles [°]: Cu-N(1') 1.9311(18), Cu-O(41) 1.9847(16), Cu-N(1) 2.0247(19), Cu-N(1")

2.0410(18), Cu-O(51) 2.1989(16), N(1')-Cu-O(41) 159.30(7), N(1')-Cu-N(1) 80.06(7), O(41)-Cu-N(1) 99.52(7), N(1')-Cu-N(1") 79.60(7), O(41)-Cu-N(1") 99.10(7), N(1)-Cu-N(1") 159.63(7), N(1')-Cu-O(51) 118.74(7), O(41)-Cu-O(51) 81.95(6), N(1)-Cu-O(51) 95.48(7), N(1")-Cu-O(51) 95.23(7).

There are π-π stacking (face-face) interactions between the molecules of the complex within the lattice. The distance between the central pyridine plane in one cation to the flanking pyridine plane in the adjacent cation is approximately 3.3 Å. (Figure 4.27). A packing diagram of the Cu(II) complex **4.7** is also shown in Figure 4.28.

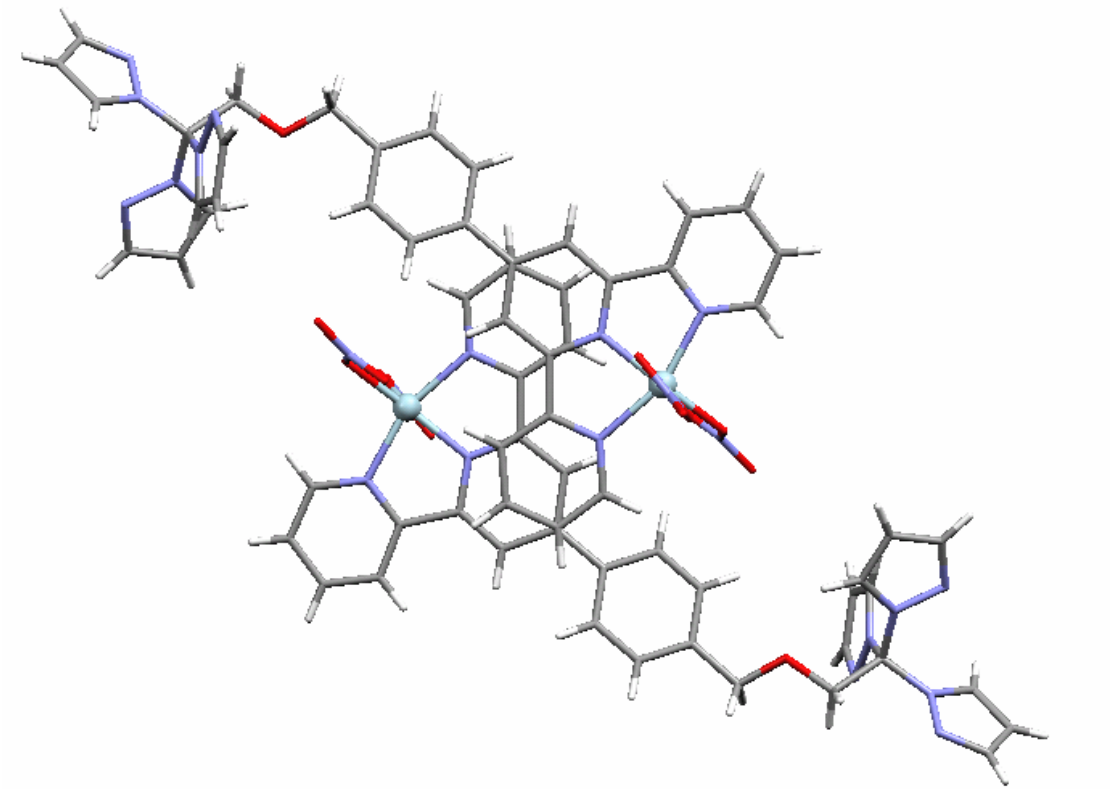


Figure 4.27. π -π stacking interaction (face-face) between the planes of the terpyridine moiety in the complex **4.7**. The separation of the central pyridine plane in one cation to the flanking pyridine plane in the adjacent cation is approximately 3.3 Å.

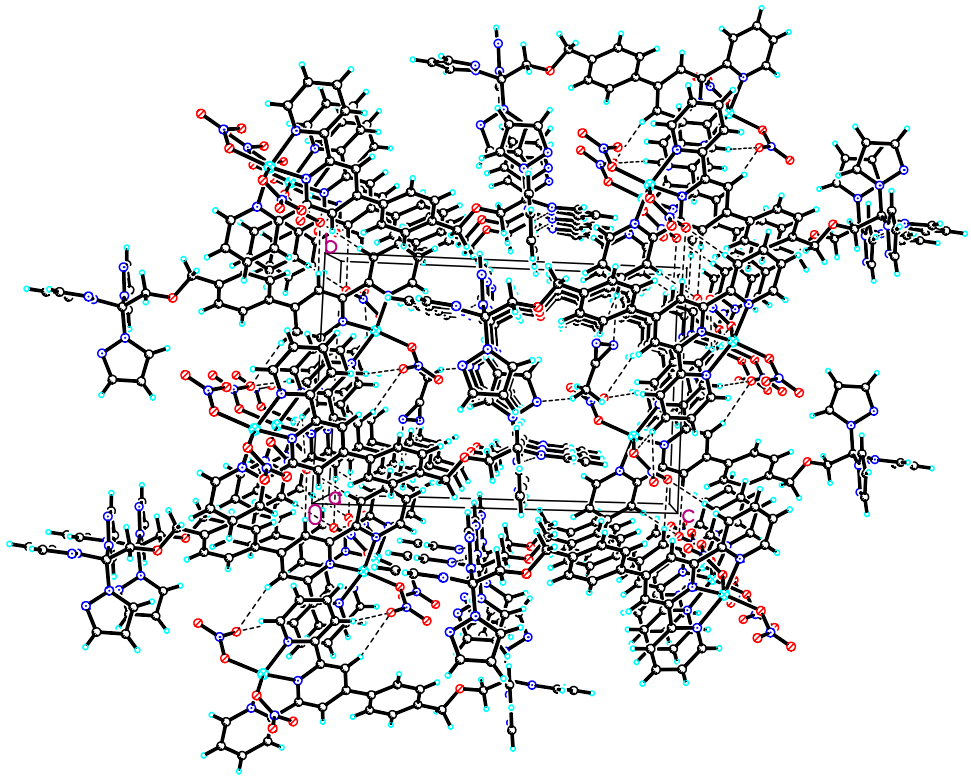


Figure 4.28. A packing diagram of the Cu(II) complex **4.7**.

4.4.4. Crystal Structure of [Zn(pzt)(Cl)$_2$], 4.8

Colourless crystals were obtained by slow evaporation of the reaction mixture from mixed solvent (CH_2Cl_2–CH_3CN) at room temperature. Compound **4.8** crystallizes in the triclinic space group, *P*$\bar{1}$. The zinc ion is coordinated to the terpyridine site, leaving pyrazolyl rings uncoordinated. The coordination geometry of zinc is approximately that of a trigonal bipyramid with N1', Cl1, and Cl2 occupying the equatorial plane and N1 and N1" in the axial positions (Figure 4.29). The principal distortion from an ideal trigonal bipyramid results from the constraints of the terpyridine ligand structure, so that the angle, at the metal, between the axial nitrogen

atom donors is only 148.54(8)º. The Cl1-Zn-N1', N1'-Zn-Cl2, and Cl2-Zn-Cl1 angles are 107.55(5), 132.68(6), and 119.75(3)º, respectively, which compare well with those for other zinc complexes based on 4'-substituted terpy systems that appear in the literature: [Zn(4'-phenyl-2,2':6'-2"-terpy)Cl_2] (105.8(1), 140.7(1), 113.5(1)º),[395] and [Zn(4'-methylthio-2,2':6'-2"-terpy)Cl_2] (106.26(10), 140.48(11), 113.25(5)º).[384] The parent structure, [Zn(2,2':6'-2"-terpy)Cl_2],[458] gives angles in the equatorial plane that are much closer to 120º (121.46(5), 121.46(5), 117.08(4)º).

The phenyl ring is nearly co-planar with the terpyridyl fragment, with the slight twist about the interannular bond such that the plane of the phenyl ring makes an angle of 4.7º with the plane of the central pyridine ring.

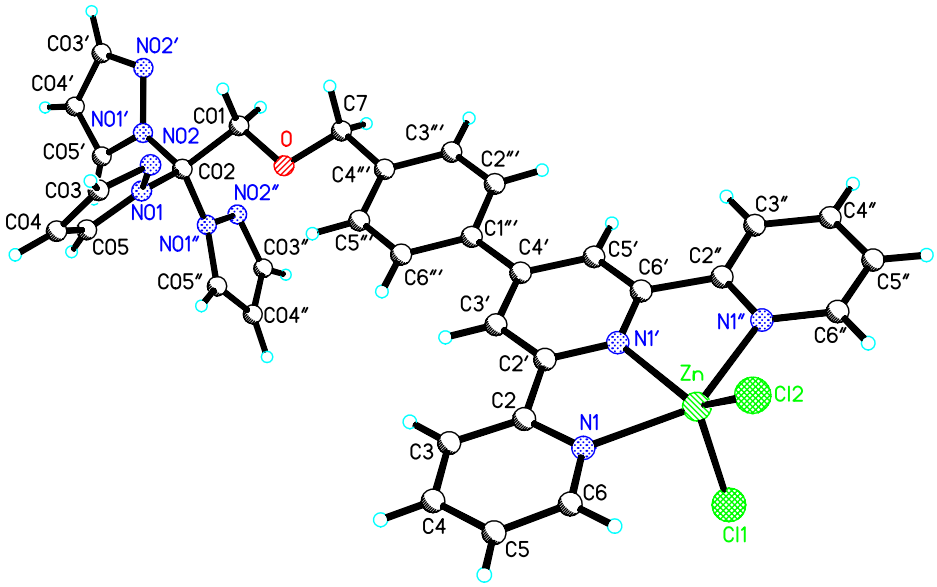


Figure 4.29. Molecular structure of the neutral Zn complex **4.8** with a CH_3CN solvated molecule omitted for clarity. Selected bond lengths [Å] and angles [°]:Zn-N(1') 2.074(2), Zn-Cl(1) 2.2704(7), Zn-N(1) 2.1818(19), Zn-N(1") 2.180(2), Zn-Cl(2) 2.2499(7), N(1')-Zn-Cl(1) 107.55(5), N(1')-Zn-N(1) 74.71(7), Cl(1)-Zn-N(1) 98.99(5), N(1')-Zn-N(1") 74.97(7), Cl(1)-Zn-N(1") 97.90(5), N(1)-

Zn-N(1") 148.54(8), N(1')-Zn-Cl(2) 132.68(6), Cl(1)-Zn-Cl(2) 119.75(3), N(1)-Zn-Cl(2) 95.63(5), N(1")-Zn-Cl(2) 98.73(5).

Again, π-π stacking interactions (face-face) are found in the lattice, and a comparison of Figures 4.27 and 4.30 reveals that the structure of the copper and zinc complexes are rather similar in this respect. Again, the plane-to-plane distances are approximately 3.3 Å (face-face, central).

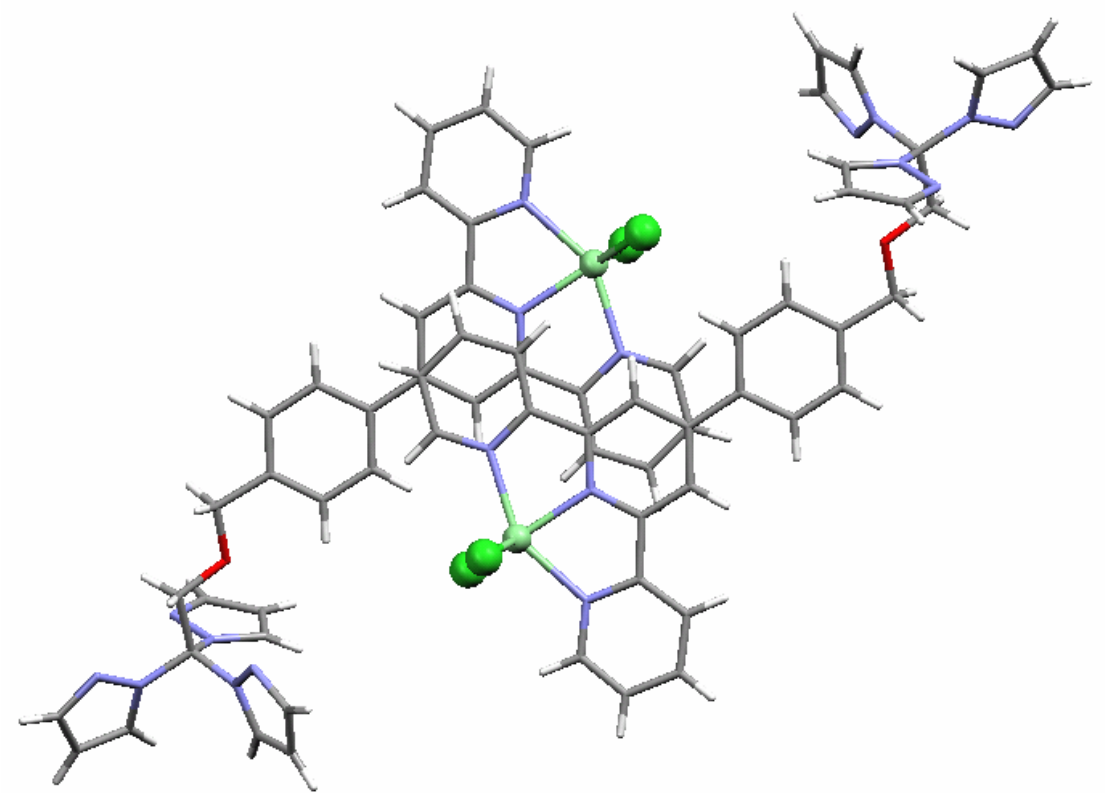


Figure 4.30. π-π stacking interaction (face-face) between the planes of the terpyridine moiety in the complex **4.8**. The separation of the central pyridine plane to the side pyridine plane is 3.3 Å.

4.5. Conclusion

The pzt ligand binds a wide range of metal ions at the preferred terpyridyl binding site but, of those metal ions that we studied, only silver(I) ions coordinate to the tris(pyrazolyl)methane binding site.

There was no evidence of any coordination to the tris(pyrazolyl) site when complexes **4.4**, **4.5**, and **4.6** were treated with $CoCl_2$, $Na_3[Co(NO_2)_6]$, or $Co(NO_3)_2$ under a variety of conditions; therefore, our strategy for preparation of a dinuclear complex containing Ru(II) and Co(III) was unsuccessful.

While the coordination chemistry of tris(pyrazolyl)methane ligands has been studied in some detail[194,202,253,262-274,283,284,396,416,417,419-423], it appears that attachment of such a group to another metal ion binding site significantly reduces its ability to bind metal ions. Presumably the positive charge on the ligand that results from coordination of a metal ion in the terpyridyl site reduces the ability of the tris(pyrazolyl)methane site to coordinate to other metal ions. We have found, however, that silver ions coordinate to both sites in the pzt ligand and a dinuclear $[Ag_2(pzt)_2]^{2+}$ complex, **4.2**, is formed when the silver ions and ligands are present in high concentration. Silver ions can also be used to link together pzt complexes of other metal ions to form polymeric species. Presumably this is a result of both the low charge on the silver ion and the ability of silver ions to bind to heterocyclic nitrogen donors.

Chapter 5

Heterodinuclear Ru(II)-Co(III) Complexes

5.1. Introduction

Ligands with macrocyclic units covalently attached to Ru(II)-terpyridyl cores, are suitable candidates for preparing heterodinuclear Ru(II)-Co(III) complexes, as the macrocycle provides a binding site for the Co(III) ion.

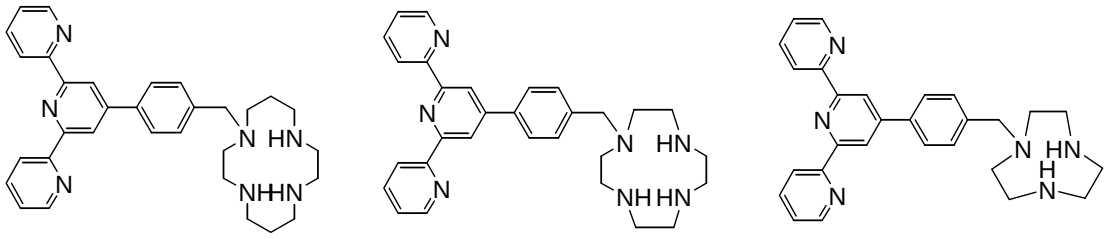
Terpyridyl systems with the appended 1,4,8,11-tetraazacyclotetradecane, cyclam macrocycle,[459] **1.74**, cymt, (Figure 5.1) have been already used as potential sensing receptors in the area of electrochemical sensor technologies.[197,460,461] Padilla-Tosta *et al.*,[197] reported that the fluorescence intensity of the Ru(II) complex comprising cyclam was quenched selectively in the presence of Cu(II) in an aqueous environment at neutral pH.

In other studies, Padilla-Tosta *et al.*[461] also demonstrated that such a Ru(II)-Cu(II) system was able to display a selective sensing response against anions such as ATP. In the presence of anions, the Ru(II)-Cu(II) system showed a fluorescent response. From the electrochemical studies, they concluded that the interaction with the anions SO_4^{2-}, PO_4^{3-}, ATP, and ADP was mainly electrostatic whereas Cl^- and Br^- formed axial covalent bonds with the Cu(II) ion in the Ru(II)-Cu(II) complex. The

addition of coordinating anions (such as Cl^-, Br^-, and OH^-) produced an enhancement of the emission intensity.

Based on potentiometric studies on the Ru(II)-Cu(II)-ATP system, they suggested that the Ru(II)-Cu(II)-ATP complex was responsible for the quenching observed at basic pH in the presence of ATP. The quenching of the intensity of the fluorescence due to ATP in the pH range 9 to 10 was selective and no other anion studied was able to quench the emission of the $Ru(tpy)_2^{2+}$ core in this pH range.

It was our intention to combine derivatised terpyridyl ligands with polyazamacrocycles (such as 1,4,8,11-tetraazacyclotetradecane[459] (cyclam), 1,4,7,10-tetraazacyclododecane[462,463] (cyclen), and 1,4,7-triazacyclononane[462,463] (tacn)), giving cymt, **1.74**, cynt, **1.75**, and tcnt, **1.76**, respectively (Figure 5.1). This will allow us systematically to develop Ru(II)-Co(III) dinuclear complexes of mixed geometry. We are also interested in reducing the number of stereoisomers that may be produced upon coordination.



1.74 **1.75** **1.76**

Figure. 5.1. Some ditopic terpyridyl-azamacrocycle ligands.

In this respect, the bridging ligand containing tacn, **1.76**, that has both a meridionally coordinating subunit (terpy) suitable for binding Ru(II) ion, and a facially coordinating triazamacrocycle (tacn) suitable for binding a Co(III) ion, seemed to be one of the most suitable candidates for our studies (Figure 5.2).

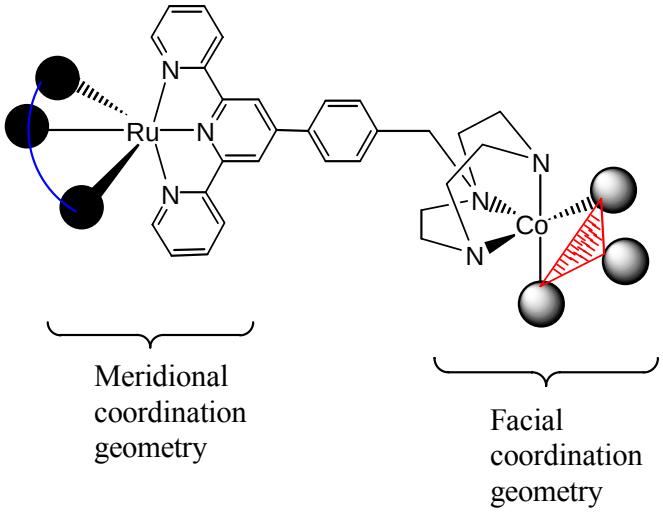


Figure 5.2. Meridional *vs.* facial coordination geometries around the octahedral environments within the ligand tcnt, **1.76**.

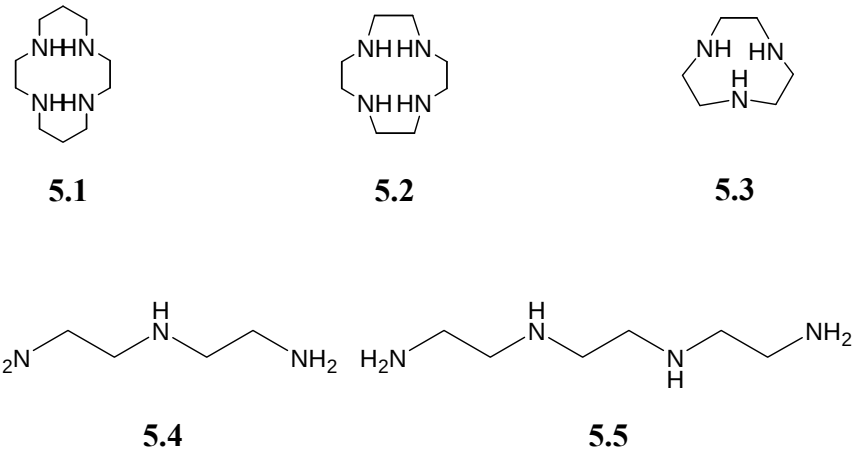
This ligand has been prepared using the literature method,[220] where the terpyridylbenzyl pendant arm is introduced in the macrocyclic structure by reaction with the tricyclic orthoamide of tacn.

Other ligands, in which a different macrocycle (cyclam/cyclen) or a polyamine (diethylenetriamine (dien)) or a mixed amine-polypyridyl (*N,N′*-bis(2-pyridylmethyl)amine,[464]) subunits have been attached to a terpyridylbenzyl pendant arm, were also considered.

This Chapter describes the synthesis, characterisation, and coordination chemistry of some new bridging ligands and their Ru(II) complexes. The synthesis and characterisation of some dinuclear Ru(II)-Co(III) complexes of a selected ligand are also described. In this chapter, our aim is to develop synthetic chemistry by which a ligand could be attached to the acceptor sites (Co(III) ion) of these systems. Such systems would be suitable to demonstrate the ligand release upon irradiation of the complexes with light.

5.1.1 Synthetic Strategies: Ligands

Selective mono-N-functionalisation of polyazamacrocycles (such as cyclam, **5.1,**[459] cyclen, **5.2**,[462,463] and tacn, **5.3**,[462,465]) and polyamines (such as dien, **5.4**, or trien, **5.5**) (Figure 5.3), is a continuing synthetic challenge, although a number of different strategies is available.[466]

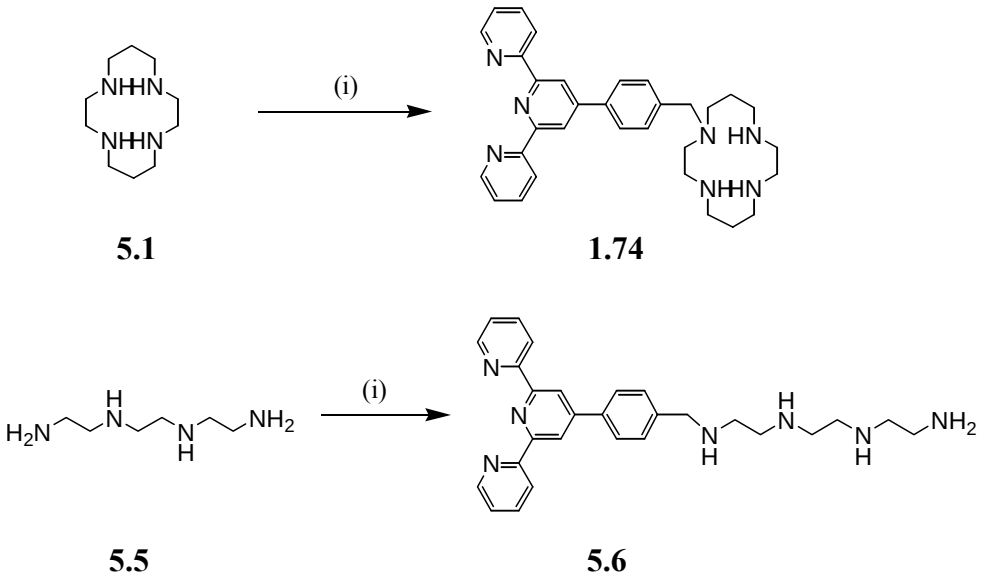


5.1 **5.2** **5.3**

5.4 **5.5**

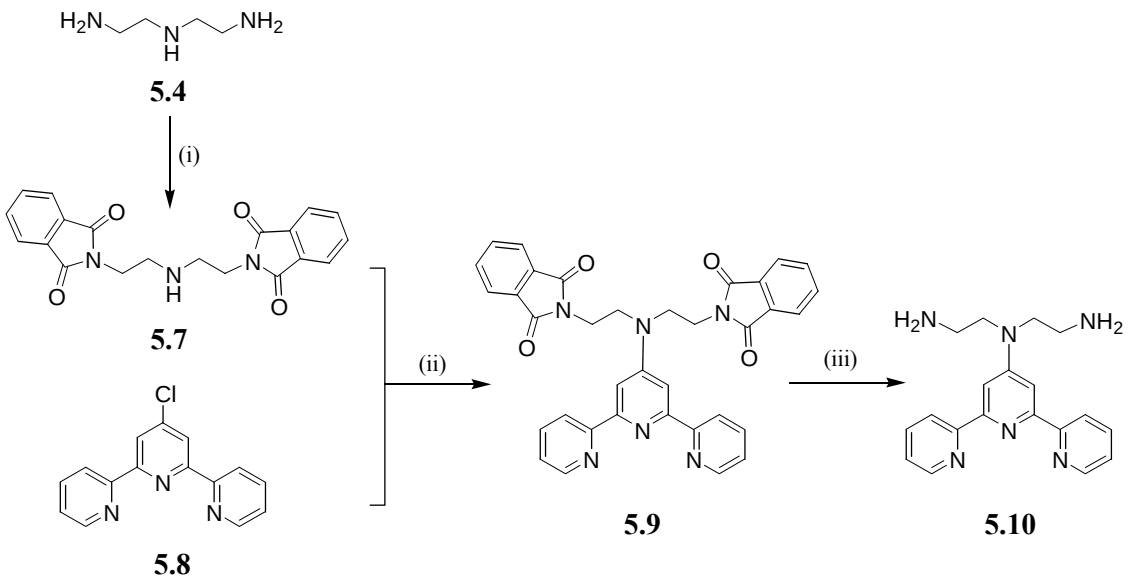
Figure 5.3. Some azamacrocycles and amines.

One common method involves direct alkylation of a large excess of macrocycle/polyamine relative to the alkyl halide in order to limit N-derivatisation to one site. This methodology has been adapted where the amine is readily available (*e.g.* in the case of cyclam, **5.1,**[197] or trien, **5.5**[467]) (Scheme 5.1). However, selective mono-N-functionalisation of cyclam using this method has not been observed, as other minor side products (such as di- and tri-substituted cyclam) could also be produced.[197] Selective functionalisation of trien, following this methodology is also problematic, as the primary/secondary amine groups within the chain are all likely to react with the reactive alkyl halide **1.50**.

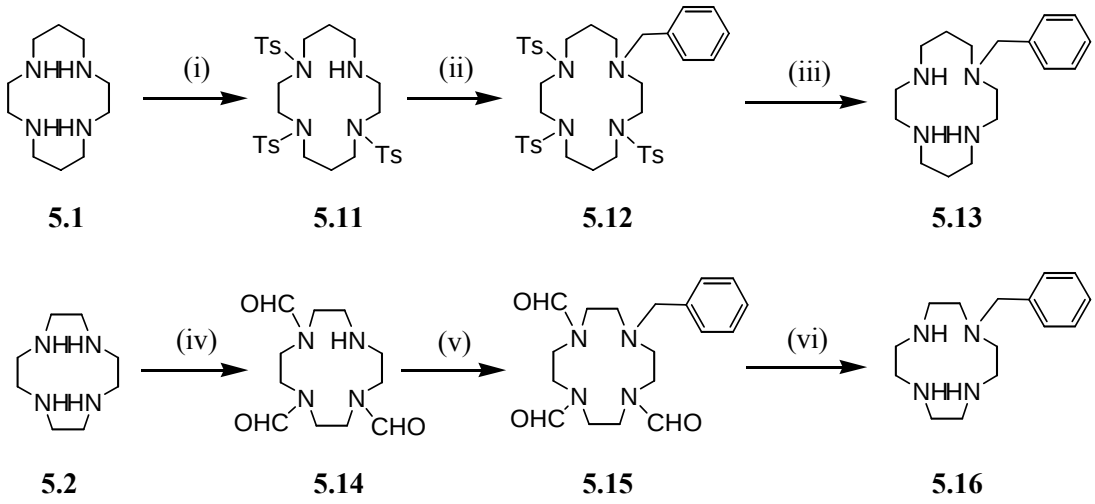


Scheme 5.1. Literature reported selective mono-N-functionalisation of polyazamacrocycles/polyamines: direct alkylation. (i) **1.50** ($^1/_5$ molar ratio), CH_2Cl_2, Et_3N, 30 ºC, 24-48 hr.[197,467]

Alternatively, protecting groups have been frequently employed to block one or more sites on the macrocycle/polyamine and so allow the remaining ones to be derivatised selectively. Although traditionally, phthalimide,[468,469] (Scheme 5.2) *tert*-butyloxycarbonyl (Boc)[470], tosyl (Ts)[471] (Scheme 5.3), or formyl (Fml)[473] (Scheme 5.3) groups have been used to protect the nitrogen atoms of the macrocycle/polyamine, new methodology with bisaminal derivatives has also been developed.[293,295,472-476]



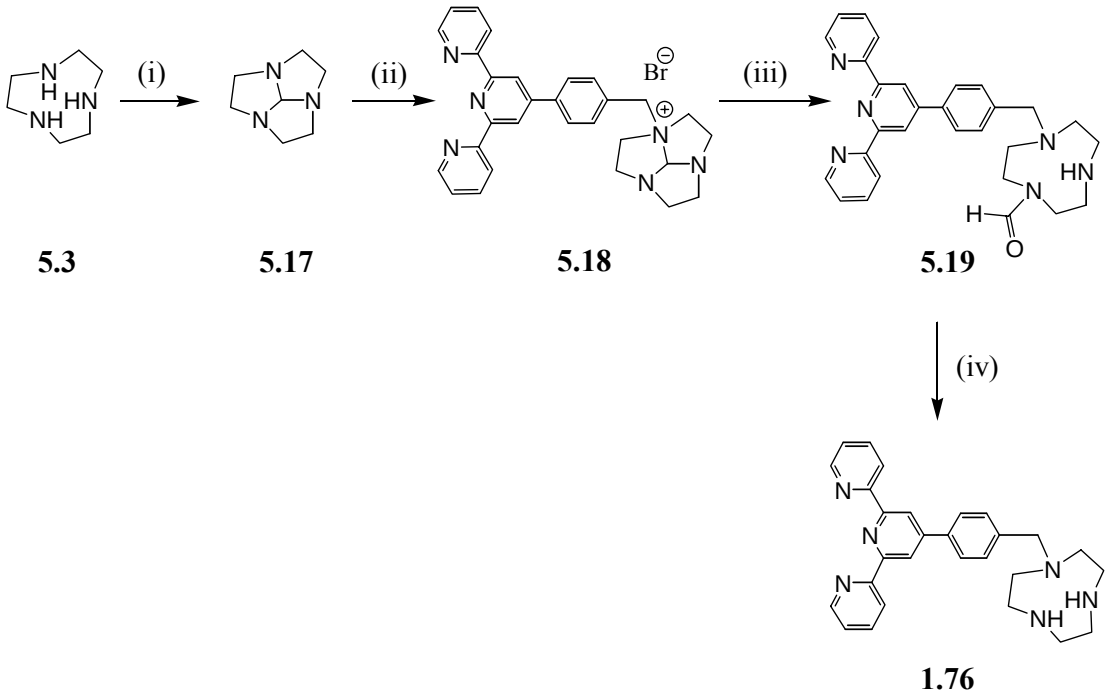
Scheme 5.2. Literature reported selective mono-N-functionalisation of dien: indirect alkylation using protection-alkylation-deprotection strategy. (i) phthalic anhydride, $CHCl_3$, reflux, 5 hr;[469] (ii) solvent-free, 180 ºC;[468] (iii) 6 M HCl, extraction.[468]



Scheme 5.3. Literature reported selective mono-N-functionalisation of polyazamacrocycles: indirect alkylation using protection-alkylation-deprotection strategy. (i) TsCl, Et_3N, CH_2Cl_2, 20 ºC, 3hr;[471] (ii) benzylbromide, $NEtPr_2^i$, EtOH, reflux, 1.5 hr;[477] (iii) conc H_2SO_4, 110 ºC, 3 days;[462] (iv) $CCl_3CH(OH)_2$, EtOH, 60 ºC;[478] (v) benzylbromide, DMF, K_2CO_3, 80 ºC;[478] (vi) 0.2 M NaOH, 80 ºC.[478]

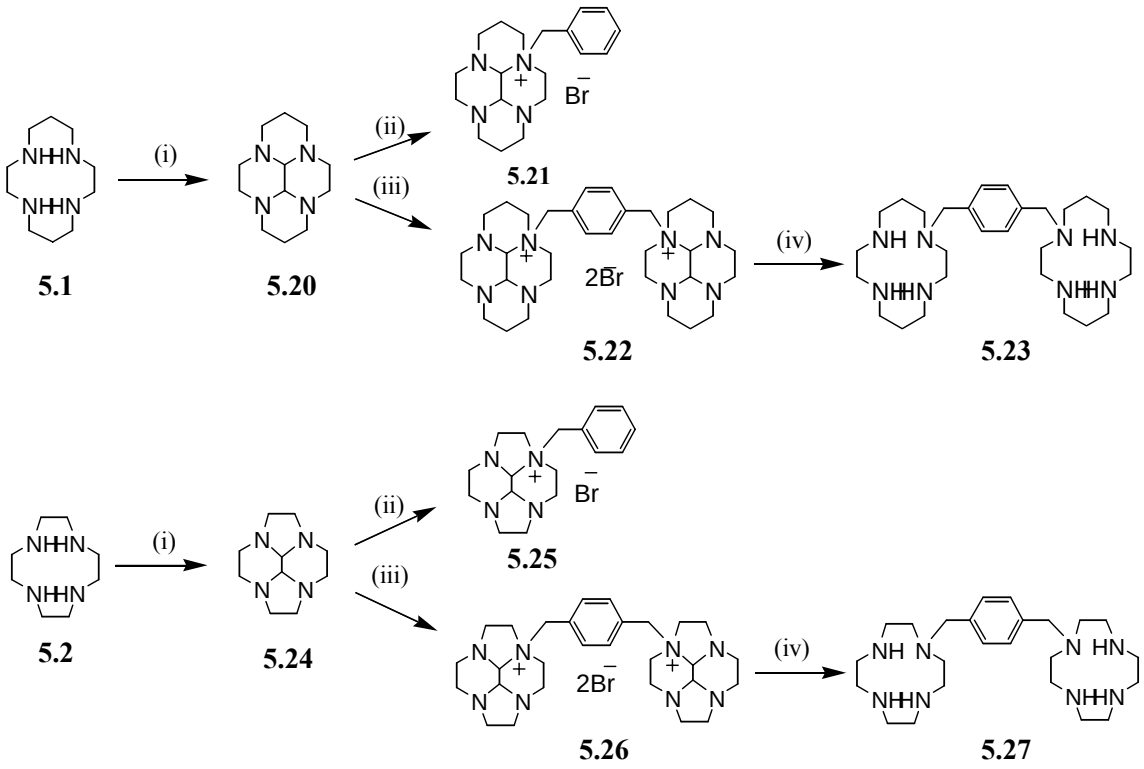
Phthalimide is normally used for protection of primary amines; while (Boc)[470], tosyl (Ts)[471] and formyl (Fml)[473] groups are used to protect secondary amines, including macrocyclic nitrogen atoms. Protection and deprotection of amine nitrogen atoms using these groups require different reaction conditions. For example, attachment of Ts groups to nitrogen atoms can be achieved in relatively mild reaction conditions[471] (normally at 25-50ºC for 3 hr); but their removal requires relatively harsh reaction conditions (conc H_2SO_4 at 110ºC for 3 days).[462] However, attachment and removal of other groups, Boc and Fml, are normally carried out in moderate reaction conditions. A catalytic hydrogenation reaction has been used to remove Boc groups in 99% yield.[470]

Synthesis of ligand tcnt, **1.76**,[220] involves (a) condensation of *N*,*N*-dimethylformamide dimethyl acetal and tacn, **5.3**,[462,465] to give "capped" 1,4,7-triazacyclononane (capped-tacn), **5.17**,[479] (b) monoalkylation of capped-tacn, **5.17**, to give a quaternary ammonium bromide **5.18**, (c) aqueous hydrolysis of **5.18** to give the aldehyde **5.19**, and (d) base hydrolysis to yield the desired ligand (Scheme 5.4).



Scheme. 5.4. Synthesis of ditopic ligand cynt, 1.75,.[220] (i) *N*,*N*-dimethylformamide dimethyl acetal, dry CH_3CN, 3 rh, 85° C;[462,465,479] (ii) **1.50** (equimolar), dry THF, 24 hr, r.t; (iii) water, reflux, 3.5 hr; (iv) KOH, EtOH:water (3:1), reflux, 48 hr.

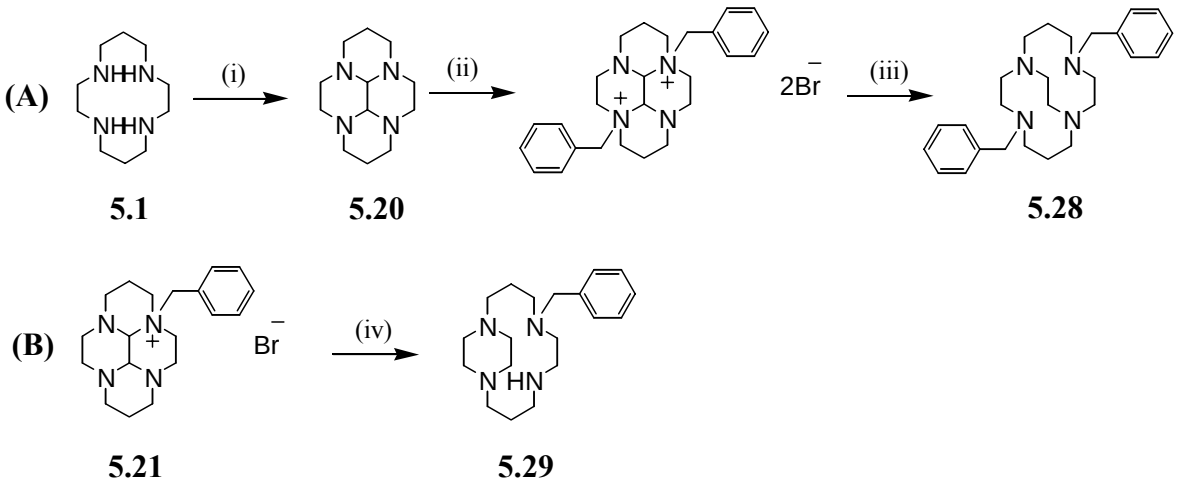
A bisaminal strategy for the synthesis of mono-N-substituted tetraazamacrocycles such as cyclen and cyclam has also been developed[480,481] (Scheme 5.5).



Scheme 5.5. Literature reported selective mono-N-functionalisation of polyazamacrocycles: bisaminal strategy.[480] (i) 40% glyoxal, MeOH; -5 to 0 ºC; r.t, 3hr; (ii) benzyl bromide, CH_3CN or THF, r.t, 2-3 hr; (iii) dibromo-*p*-xylene, CH_3CH, r.t, 24 hr; (iv) H_2NNH_2, H_2O, reflux, overnight.

Wong *et al.*,[295] Weisman *et al.*,[293] and Le Baccon *et al.*,[480] have also reported the syntheses of symmetrical and asymmetrical bis-N1,N3-functionalised tetraazamacrocycles using bisaminal methodology. Their short, efficient route to the cross-bridged cyclams and selected pendant-arm derivatives is shown in Scheme 5.6.

Koliński[482] has studied the ring cleavage reduction reactions of mono-N-substituted bisaminal cyclam. They have established that when mono-functionalised **5.21** was subjected to reduction using $NaBH_4$, piperazinocyclam **5.29** was obtained (Scheme 5.6). There was no indication of formation of mono-substituted cross-bridged cyclams using this approach.



Scheme 5.6. Literature reported selective mono-N- and bis-N1,N3-functionalisation of cyclam: bisaminal strategy. **(A)**[295] (i) 40% glyoxal, MeOH; -5 to 0 ºC; r.t, 3hr; (ii) excess benzyl bromide, CH_3CN, r.t, 14 days; (iii) $NaBH_4$, 95% EtOH, r.t, 16 days. **(B)**[482] (iv) $NaBH_4$, 96% EtOH, r.t, 5 days, under Ar.

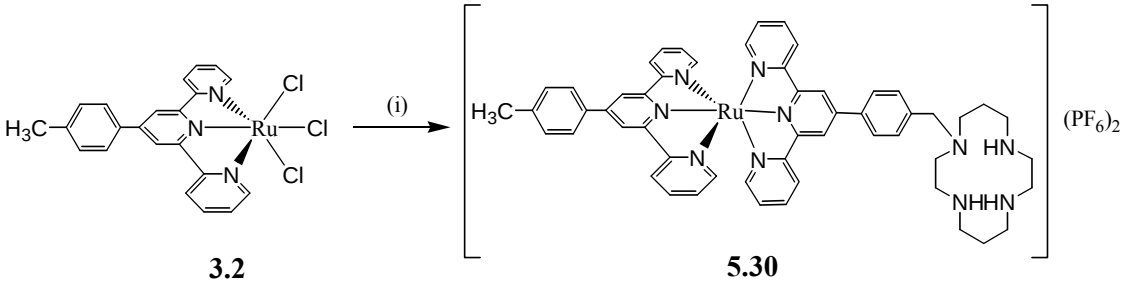
Compound **5.29** and its analogues produce complexes with *trans* isomers around octahedral metal centres; therefore they might be useful for our studies, as the number of possible stereoisomers of octahedral complexes containing piperazinocyclams are limited.

5.1.2. Synthetic Strategies: Complexes

To synthesise the Ru(II) complexes, where Ru(II) ions are bound into the terpyridyl cores of the ditopic ligands selectively, two main approaches are possible: (1) "direct complexation" and (2) "protection-complexation-deprotection".

"Direct complexation" involves the direct reaction of the Ru(II) complex precursor **3.2** with the ditopic ligands in one step. Since the Ru(II) ion in the complex precursor **3.2** is already meridionally coordinated, it should prefer coordination to the

meridionally coordinating subunit/s of the ligands. This method can be applied to the ditopic ligands where the two metal ion binding sites are differentiated either by the number of donor atoms in each site, or by the configuration of the binding site (*e.g.* meridional *vs.* facial geometries). This kind of approach has been used before, and a range of Ru(II) complexes have been prepared selectively. For example, Padilla-Tosta *et al.*[197] have prepared the Ru(II) complex **5.30** (containing cymt) (Scheme 5.7) in which the ligand contains two different binding sites: a meridionally tpy binding domain and a tetraazamacrocyle (cyclam).



3.2 **5.30**

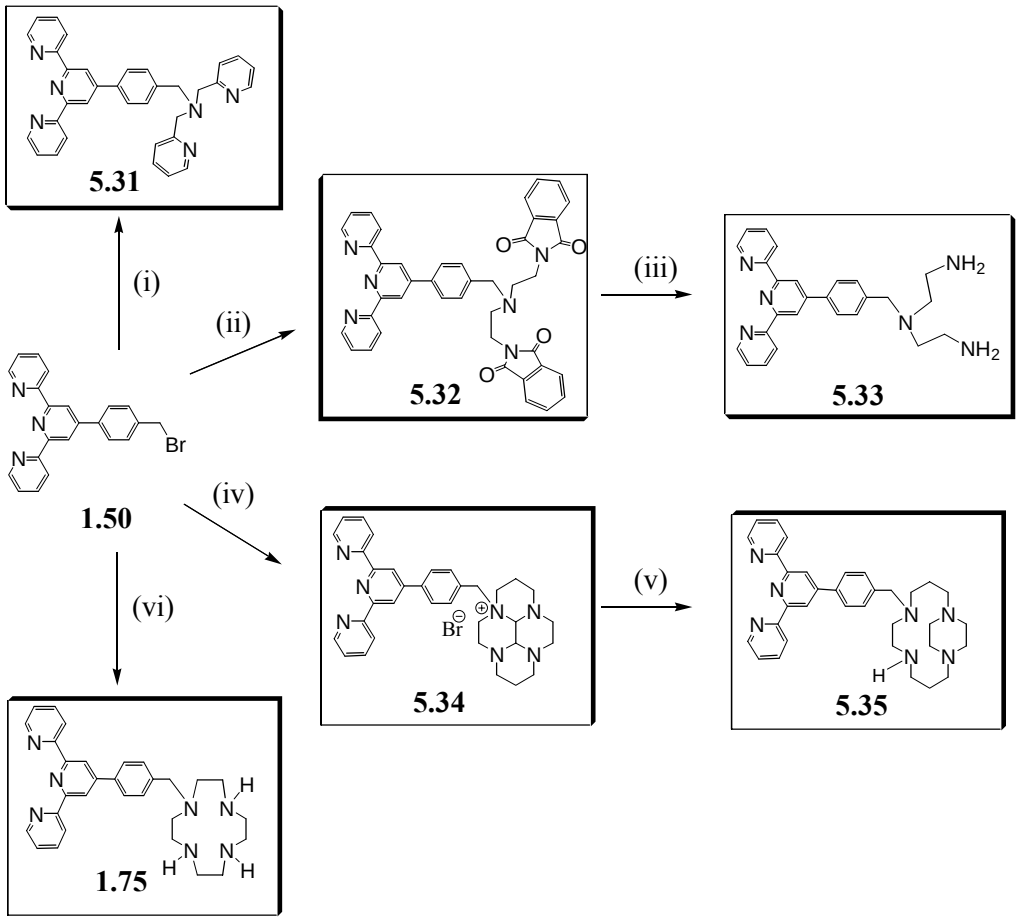
Scheme 5.7. Synthesis of a Ru(II) complex containing ditopic ligand cymt, **1.74**. (i) cymt, **1.74**, *N*-ethylmorpholine, MeOH, reflux, 2 h; NH_4PF_6.

When the ligand contains similar binding sites, then a "protection-complexation-deprotection" method would be a better choice. In this method, the other site of the ligand is blocked in such a way that only the meridionally coordinating subunit (tpy) could coordinate to the Ru(II) complex precursor **3.2** selectively. This kind of approach has also been used before, and a number of complexes were prepared selectively. For example, Woodhouse and Rendina[483] have reported on the synthesis of some multinuclear Pt(II)–amine complexes selectively. Robillard *et al.*[484] also used this method to prepare some mononuclear tetraamineplatinum (II) complexes

selectively. This approach may use the protecting groups introduced during ligand synthesis to direct the complexation to form the desired products.

5.2. Results and Discussion

5.2.1. Ligands Syntheses



Scheme 5.8. Syntheses of new ligands. (i) dipicolylamine (*N*,*N*′-bis(2-pyridylmethyl)amine),[464] K_2CO_3, dry CH_3CN, 3 days, 60º C; (ii) 1,5-bis(phthalimido)-3-azapentane,[469] K_2CO_3, dry CH_3CN, 3 days, 60º C; (iii) ethylenediamine, MeOH, over night, r.t; (iv) bisaminal-cyclam, **5.20**, dry THF, 4 days, r.t; (v) $NaBH_4$, EtOH, r.t, 16 days; (vi) **5.2**, dry CH_2Cl_2, triethylamine, 24 hr, 30° C.

Three new ditopic ligands 4'-(*p*-(1,5-bis(phthalimido)-3-azapentan-3-yl)methylphenyl)-2,2':6',2"-terpyridine (bpat), **5.31**, 4'-(*p*-(1,5-bis(amino)-3-azapentan-3-yl)methylphenyl)-2,2':6',2"-terpyridine (dint), **5.33**, and 4'-(*p*-(1,4,7,10-tetraazacyclododec-1-yl)methylphenyl)-2,2':6',2"-terpyridine (cynt), **1.75**, based on tridentate planar terpyridyl systems (Scheme 5.8) have been synthesised in relatively high yields. A new intermediate 4'-(*p*-(10bα,10cα)-decahydro-3a-1*H*,6*H*-3a,5a,8a,10a-tetraazapyrenium-methylphenyl)-2,2':6',2"-terpyridine bromide (ptmtb), **5.34**, and a new ligand 4'-(*p*-(1,5,8,12-tetraazabicyclo[10.2.2]hexadec-5-yl-methylphenyl)-2,2':6',2"-terpyridine (pcymt), **5.35**, have also been synthesised in low yield. The ligands shown in Scheme 5.8 were all prepared using the above mentioned methods by nucleophilic substitution reaction of 4′-(*p*-bromomethylphenyl)-2,2′:6′,2″-terpyridine, **1.50**, with the appropriate secondary or tertiary amines in single- or multi-step reactions.

Ligand bpat, **5.31** was synthesised using direct functionalisation of dipicolylamine (*N*,*N*′-bis(2-pyridylmethyl)amine)[464] with the bromo compound **1.50** (Scheme 5.8). The synthetic methodology shown in Scheme 5.2 was adapted for synthesis of ligand **5.2** (Scheme 5.8) by reacting of 1,5-bis(phthalimido)-3-azapentane, bpta, **5.7**,[469] with the bromo compound **1.50** (Scheme 5.8). The reactions were carried out in dry CH_3CN and in the presence of K_2CO_3 to afford the desired products in excellent yields (95-98%). The reaction mixtures were monitored by ^1H NMR spectroscopy and thin layer chromatography (TLC) in order to determine the optimum conditions. It was noted that prolonged reactions (up to 3 days) and also using 1.2 equivalents of the amines resulted in analytically pure products, with the best yields. The ligand bptt, **5.32**, was then converted to ligand dint, **5.33** by deprotection using ethylenediamine, in MeOH, in nearly quantitative yield. All products were

characterised using ^{1}H and ^{13}C NMR spectroscopy, COSY, NOESY, HSQC, and electrospray ionisation mass spectrometry.

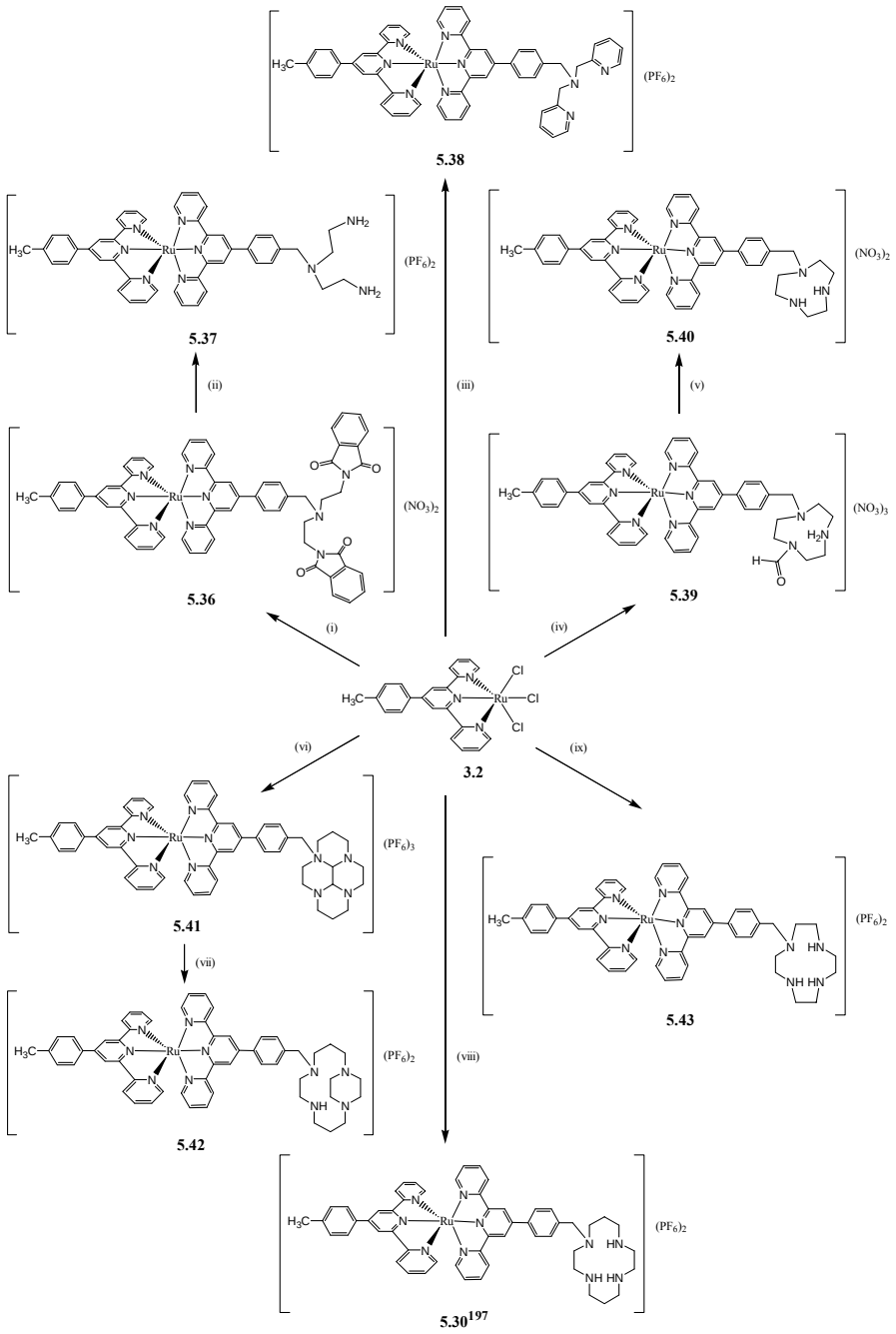
Since azamacrocycles cyclam and cyclen are readily available, the single-step "direct functionalisation" method was used to synthesise the ligand cymt, **1.74**,[197] and the new ligand cynt, **1.75**, (Scheme 5.8), respectively. The excess macrocycles were recovered and reused. We have, however, found that the choice of solvent is essential for avoiding di- or tri-alkylation, as well as for recovery of the excess macrocycle. We have observed that using toluene in the case of cymt, **1.74**, not only afforded the product in high yield, but also recovery of excess cyclam was more efficient than when other solvents (CH_2Cl_2,[197] CH_3CN or THF) were used. Similarly, CH_2Cl_2 was the best solvent in which the synthesis of cynt, **1.75**, was carried out.

Bisaminal methodology[295,480,485] was employed for synthesis of ligand pcymt, **5.35**, (Scheme 5.8). We have found that mono-N-substituted ptmtb, **5.34**, was the key intermediate in the synthesis of pcymt, **5.35**. The reaction of the bisaminal-cyclam, **5.20**, with the bromo compound **1.50** was carried out in a variety of conditions (different solvents such as CH_3CN, toluene, or THF and different time scales, two hours to two weeks); however, the yields never exceeded 11% and needs improvement in the future.

5.3. Syntheses of Complexes

Ru(II) complexes **5.36** (containing bptt), **5.38** (containing bpat), **5.39**, **5.41**, **5.30** (containing cymt),[197] and **5.43** (containing cynt) (Scheme 5.9) were synthesised by reaction of the ruthenium complex precursor [Ru(ttp)Cl_3], **3.2**, with the corresponding ligands in boiling MeOH. All crude samples of complexes were

collected as their PF_6^- salts. Further purification of the complexes were achieved either using column chromatography (silica gel, eluting with CH_3CN/saturated KNO_3/water) or by recrystallisation of the complexes. Methanol soluble NO_3^- salts **5.36** (containing bptt) and **5.39** were used for the subsequent reactions to synthesise complexes **5.37** (containing dint) and **5.40** (containing tcnt), respectively (the "deprotection" step). All Ru(II) complexes were characterised using ^{1}H NMR, ^{13}C NMR, COSY, NOESY, and HSQC techniques as well as electrospray ionisation mass spectrometry, UV-vis spectroscopy, and elemental analysis.



Scheme 5.9. Syntheses of complexes. (i) bptt, **5.32**, *N*-methylmorpholine, MeOH, reflux, 2 h (ii) ethylenediamine, MeOH, r.t, overnight; (iii) bpat, **531**, *N*-methylmorpholine, MeOH, reflux, 2 h; NH_4PF_6 (iv) **5.18**, *N*-methylmorpholine, MeOH, reflux, overnight; (v) KOH, EtOH:water (3:1), reflux, 3 days; (vi) ptmtb, **5.34**, *N*-methylmorpholine, MeOH, reflux, overnight; NH_4PF_6 (vii) $NaBH_4$, 95% EtOH, 14 days, r.t; (viii) cymt, **1.74**, *N*-methylmorpholine, MeOH, reflux, 2 h; NH_4PF_6; (ix) cynt, **1.75**, *N*-methylmorpholine, MeOH, reflux, 2 h; NH_4PF_6.

As shown in Scheme 5.9, Ru(II) complexes **5.37** (containing dint) and **5.40** (containing tcnt) were prepared using "protection-complexation-deprotection" methodology in relatively good yields and purities. We have found that the reactions of the deprotected ligands bpat, **5.31**, dint, **5.33**, and tcnt ,**1.76**, with the Ru(II) complex precursor **3.2**, to synthesise the corresponding complexes **5.38**, **5.37** and **5.40** using the "direct complexation" approach were problematic. TLC of the reaction mixtures suggested that many products were probably formed during the reactions. Ligands bpat, **531**, and dint, **5.33**, have three N donor atoms at each site. The amine and the picolylamine binding sites in bpat, **531**, and dint, **5.33**, respectively, may also react with Ru(II) complex **3.2** in a meridionally fashion to produce undesired side products.

However, the two-step reaction approach (Scheme 5.9) led us to obtain the desired product, **5.37**, in higher yields (90% *vs.* 10%) with successful purification using standard column chromatography on silica gel.

We have also observed that synthesis of complex **5.40** (containing tcnt) using "protection-complexation-deprotection" method was more efficient (50% *vs.* 5%) than when the "direct complexation" method was applied.

Ru(II) complex **5.43** (containing cynt) was synthesised in a similar fashion as described for preparation of the complex **5.30** (containing cymt)[197] using the "direct complexation" approach.

In order to reduce the number of the stereoisomers that may produced upon reaction of Ru(II) complex **5.30** with Co(III) ions, synthesis of Ru(II) complex **5.42** (containing pcymt) using "protection-complexation-deprotection" approach was attempted. The intermediate **5.41** was prepared in good yield and purity; but the overall synthesis is hampered by the poor yield of the ligand ptmtb, **5.34**.

Unfortunately, deprotection of the product by reductive ring cleavage using $NaBH_4$ in 95% EtOH[295] did not go to completion (even after four weeks) and purification of the crude material using column chromatography on silica was also unsuccessful.

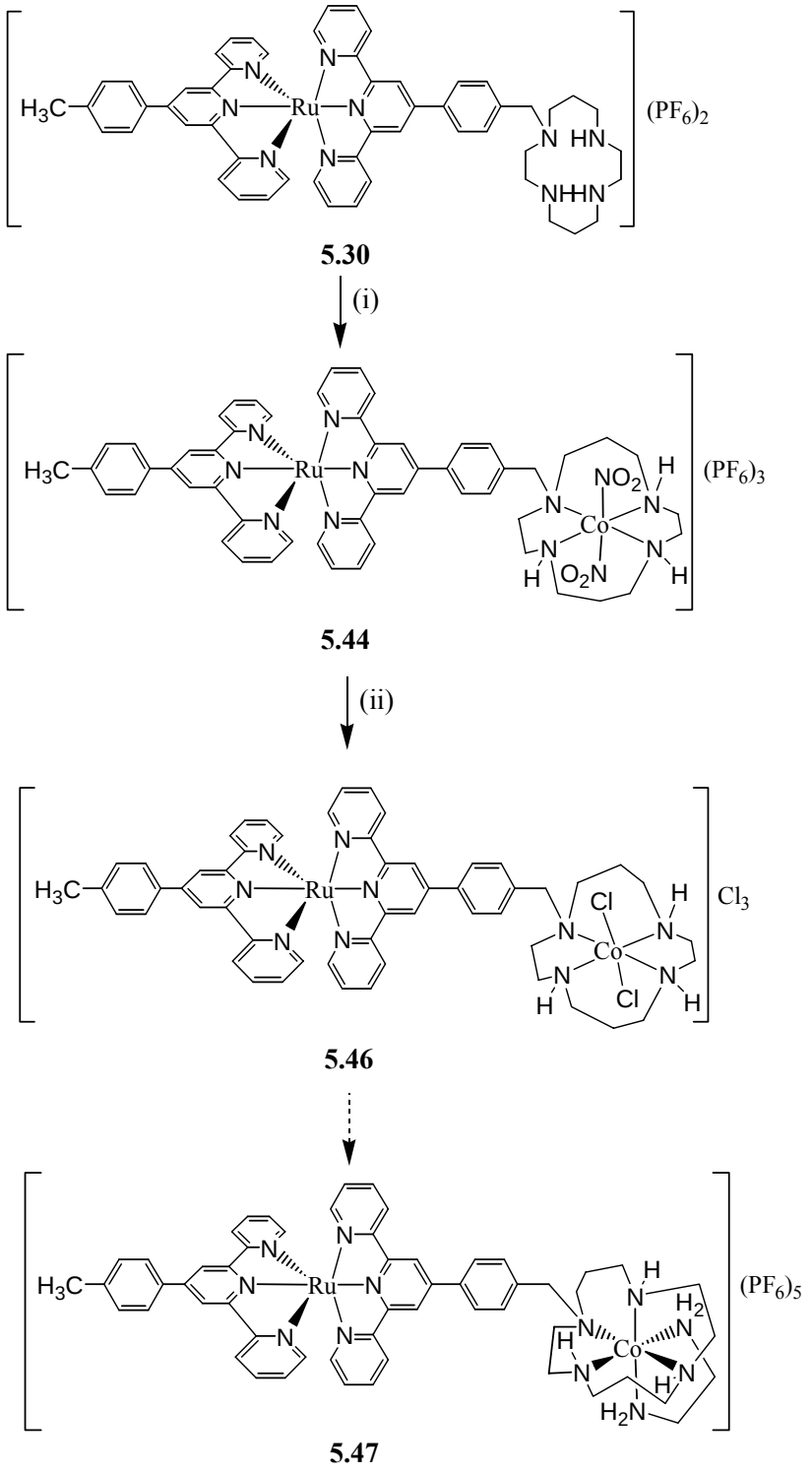
5.2.3. Heterodinuclear Ru(II)-Co(III) Complexes

Heterodinuclear complex **5.44** was prepared by reacting the Ru(II) complex **5.30** in acetone with an equimolar amount of $Na_3[Co(NO_2)_6]$ in water at 50ºC in the presence of triethylamine (Scheme 5.10). The product was collected as its PF_6^- salt and its composition was characterised using ^1H NMR, ^{13}C NMR, COSY, NOESY, and HSQC techniques as well as ESI-MS, UV-vis spectroscopy, and elemental analysis. Unfortunately, attempts to obtain single crystals suitable for X-ray crystallography to confirm which isomer was obtained, proved unsuccessful.

We are aware of literature reports[291,486] in which the authors suggested that only *trans* isomers of Co(III) complexes with cyclam and other monodentate ligands (such as NO_2^-, Cl^-, NCS^-, or Br^-) in aqueous solutions can be obtained. Poon and Tobe[487] also reported that *cis* complexes with monodentate ligands in aqueous solution eventually isomerise to a *trans* configuration. According to Bosnich *et al*,[291] in none of the preparations starting from *trans*-$[Co(cyclam)(NO_2)_2]^+$ or *trans*-$[Co(cyclam)(Cl)_2]^+$, is there any indication of the presence of a second component that is isomeric with the main *trans* crop.

When the similar reaction was carried out in the presence of NaOH, a dinuclear complex, **5.45**, was obtained, after purification using column chromatography, where two OH^- anions were coordinated to the Co(III) ion in the cyclam cavity (Scheme 5.10). The ^1H NMR spectrum of this product exhibits broad signals (Figure 5.4). The

^{1}H NMR spectra of the complexes **5.30** and **5.44** are also shown in Figure 5.4 for comparison. It is not clear, at this stage, why the spectrum of **5.45** is so much broader.



Scheme 5.10. Syntheses of heterodinuclear Ru(II)-Co(III) complexes. (i) $Na_3[Co(NO_2)_6]$, acetone/water (1:2), 50 ºC, overnight, Et_3N; (ii) 6M HCl, steam bath 8 hr.

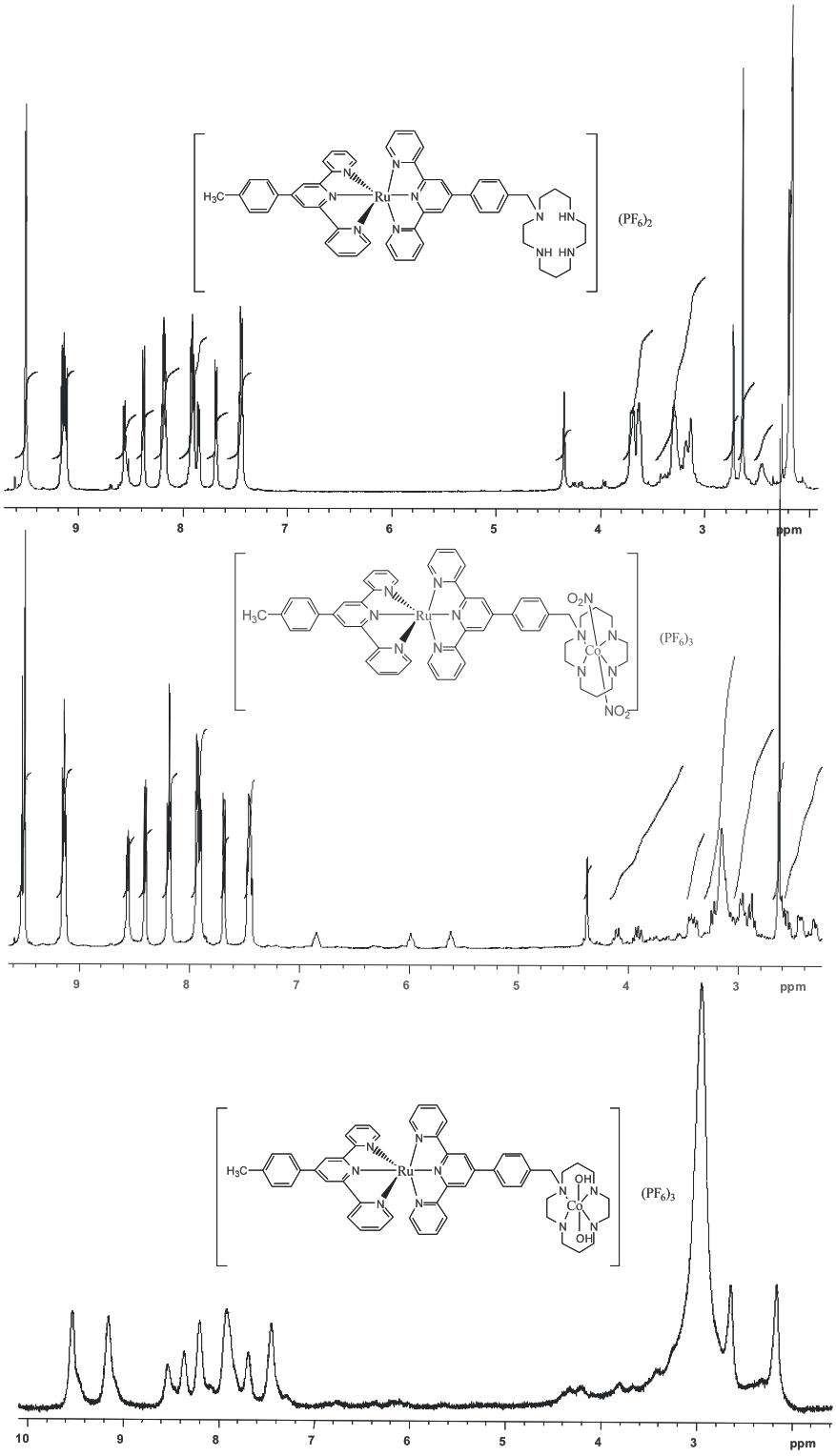


Figure 5.4. ^{1}H NMR spectrum of the Ru(II) complexes in acetone-d_6 Top: [(ttp)Ru(cymt)]$(PF_6)_2$, **5.30**; middle: [(ttp)Ru(cymt)Co$(NO_2)_2$]$(PF_6)_3$, **5.44**; below [(ttp)Ru(cymt)Co$(OH)_2$]$(PF_6)_3$, **5.45**.

The complex **5.44** was isolated as its PF_6^- salt in 84% yield. ^{1}H NMR, ^{13}C NMR, electrospray mass spectra, and elemental analysis of the complex were all consistent with the proposed structure. Subsequently, treatment of the complex **5.44** with 6M HCl on the steam bath resulted the complex **5.46** (Scheme 5.10). Complex **5.46** was also characterised using ^{1}H NMR, ^{13}C NMR, and ESI-MS techniques. Electrospray mass spectra of the complexes **5.44** (*m/z* 365.8482 and 621.2722 corresponding to $[(ttp)Ru(cymt)Co(NO_2)_2]^{3+}$ and $[(ttp)Ru(cymt)Co(NO_2)_2+(PF_6)]^{2+}$, respectively) and **5.46** (*m/z* 358.4726 and 556.2232 corresponding to $[(ttp)Ru(cymt)Co(Cl)_2]^{3+}$ and $[(ttp)Ru(cymt)Co(Cl)_2+Cl]^{2+}$, respectively) are shown in Figures 5.6 and 5.7, respectively. The ESI-MS data (*m/z* 315.80, 473.28, and 546.26, corresponding to $[(ttp)Ru(cymt)+H]^{3+}$, $[(ttp)Ru(cymt)]^{2+}$, and $[(ttp)Ru(cymt)+H+(PF_6)]^{2+}$, respectively) for the Ru(II) complex **5.30** are shown in Figure 5.5 for comparison.

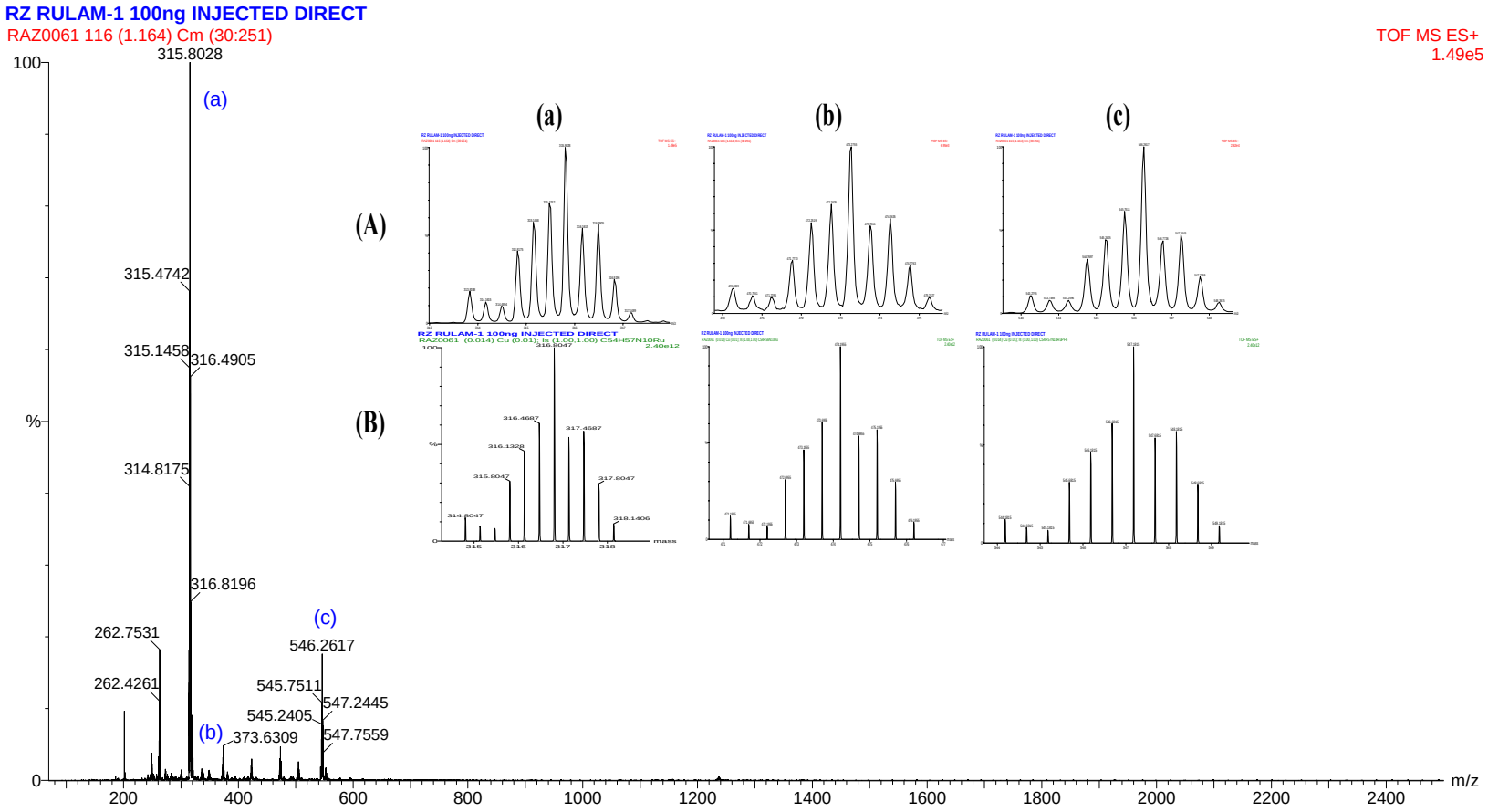


Figure 5.5. ESI-MS of $[(ttp)Ru(cymt)](PF_6)_2$ complex, **5.30**, in CH_3CN. (A) High resolution scans of the two major signals (a), (b), and (c); (B) Calculated isotope distribution patterns for (a) $[(ttp)Ru(cymt)+H]^{3+}$, (b) $[(ttp)Ru(cymt)]^{2+}$, and (c) $[(ttp)Ru(cymt)+H+(PF_6)]^{2+}$ (c) at *m/z* 315.80, 473.28, and 546.26, respectively.

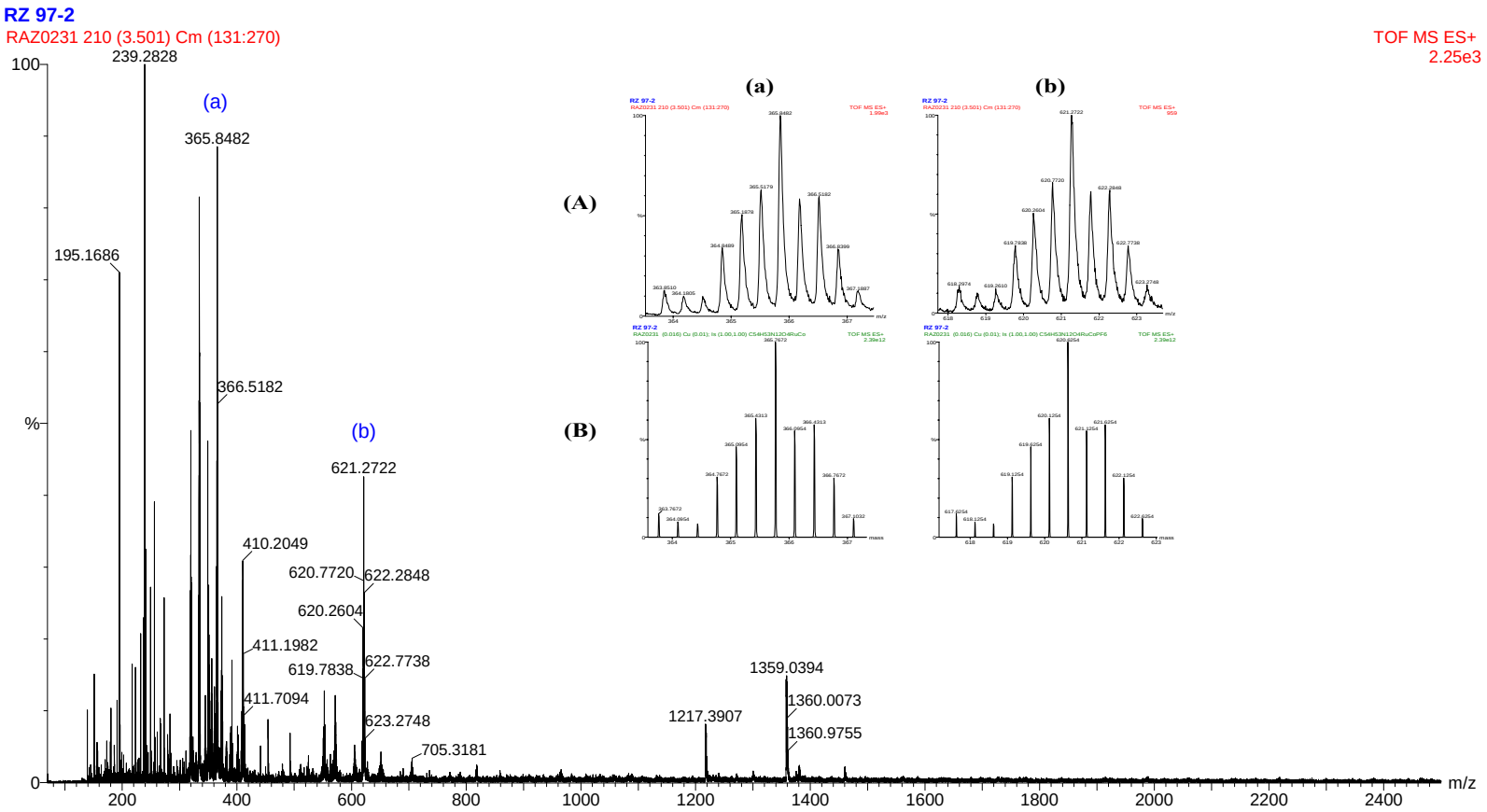


Figure 5.6. ESI-MS of $[(ttp)Ru(cymt)Co(NO_2)_2](PF_6)_3$ complex, **5.44**, in CH_3CN. (A) High resolution scans of the two major signals (a), and (b); (B) Calculated isotope distribution patterns for (a) $[(ttp)Ru(cymt)Co(NO_2)_2]^{3+}$ and (b) $[(ttp)Ru(cymt)Co(NO_2)_2{+}(PF_6)]^{2+}$, at *m/z* 365.8482 and 621.2722, respectively.

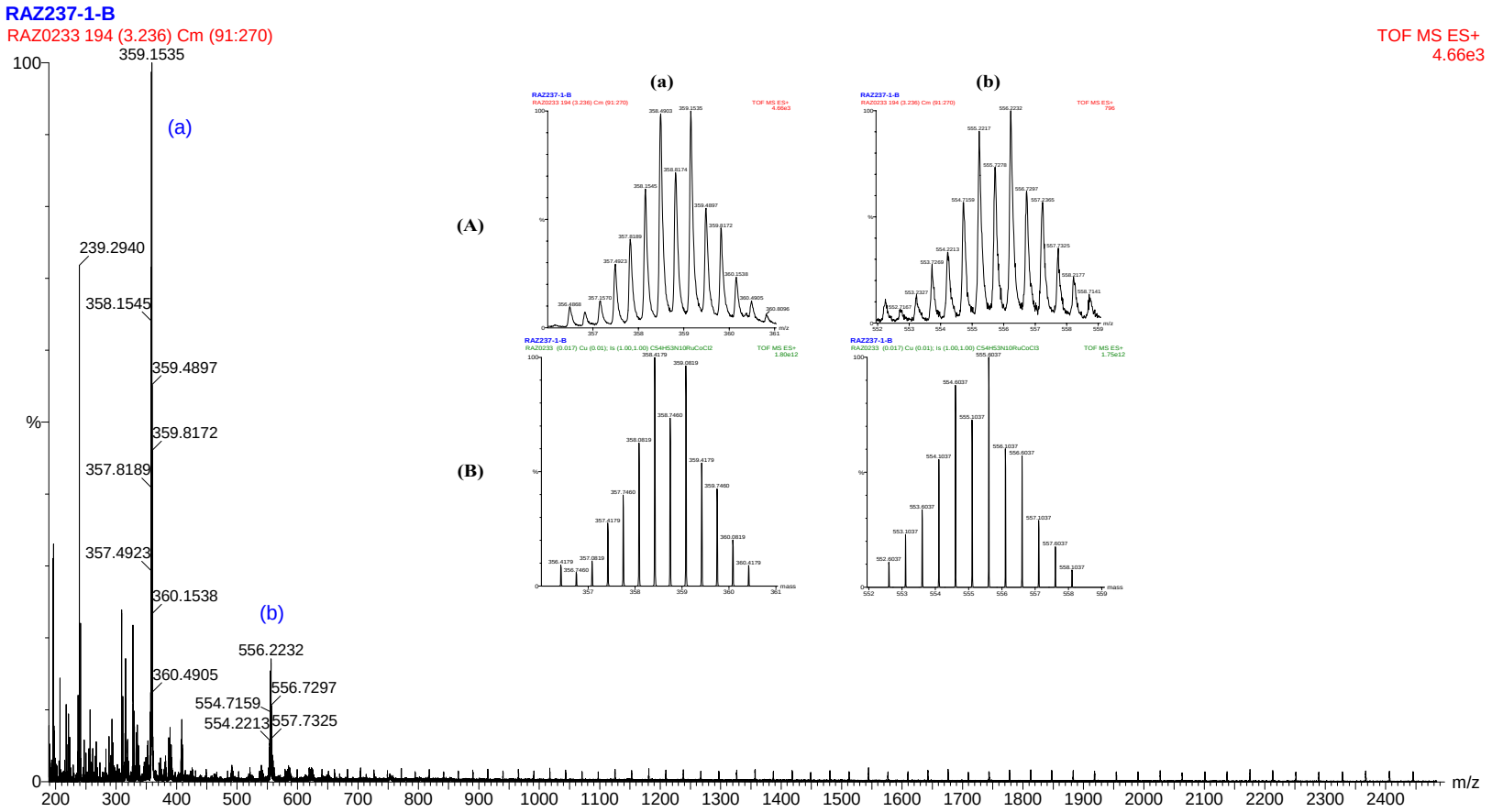


Figure 5.7. ESI-MS of $[(ttp)Ru(cymt)Co(Cl)_2]Cl_3$ complex, **5.46**, in CH_3CN. (A) High resolution scans of the two major signals (a), and (b); (B) Calculated isotope distribution patterns for (a) $[(ttp)Ru(cymt)Co(Cl)_2]^{3+}$ and (b) $[(ttp)Ru(cymt)Co(Cl)_2{+}Cl]^{2+}$, at *m/z* 358.4726 and 556.2232, respectively.

The UV-vis spectra of **5.30**, **5.44**, and **5.46** were, as expected, very similar in appearance (Figure 5.8) as they are dominated by the MLCT transitions of the Ru(II) polypyridyl complexes which have much higher molar extinction coefficients than the d-d transitions for Co(III) amine complexes.[488] The λ_{max} for each of these complexes at 490 nm corresponds to MLCT bands, where M is Ru(II).

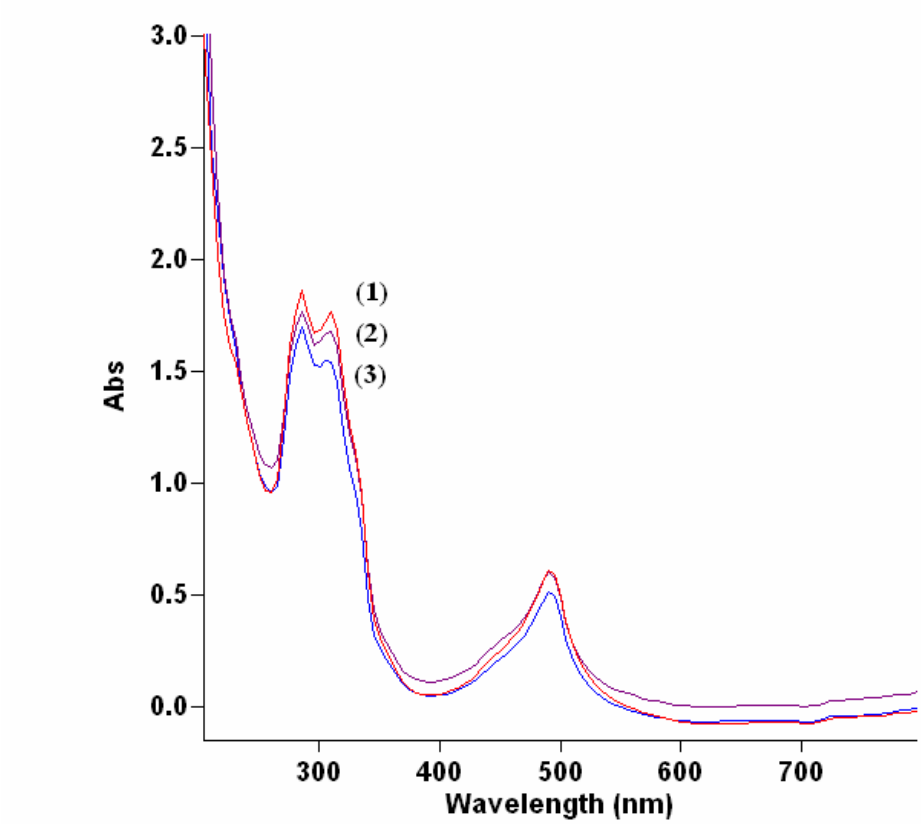


Figure 5.8. UV-vis spectra of: **(1)** $[(ttp)Ru(cymt)Co(NO_2)_2](PF_6)_3$, **5.44, (2)** $[(ttp)Ru(cymt)Co(Cl)_2]Cl_3$, **5.46**, and **(3)** $[(ttp)Ru(cymt)](PF_6)_2$, **5.30**.

The ESI-MS of $[(ttp)Ru(cymt)Co(OH)_2](PF_6)_3$, **5.45**, exhibits peaks at *m/z* 346.80 and 519.18, that can be assigned to the $[(ttp)Ru(cymt)Co(OH)_2]^{3+}$ and $[(ttp)Ru(cymt)Co(OH)_2{+}(PF_6)]^{2+}$ cations, respectively, with the observed isotope patterns correlating well with those predicted for the complex ions (Figure 5.9).

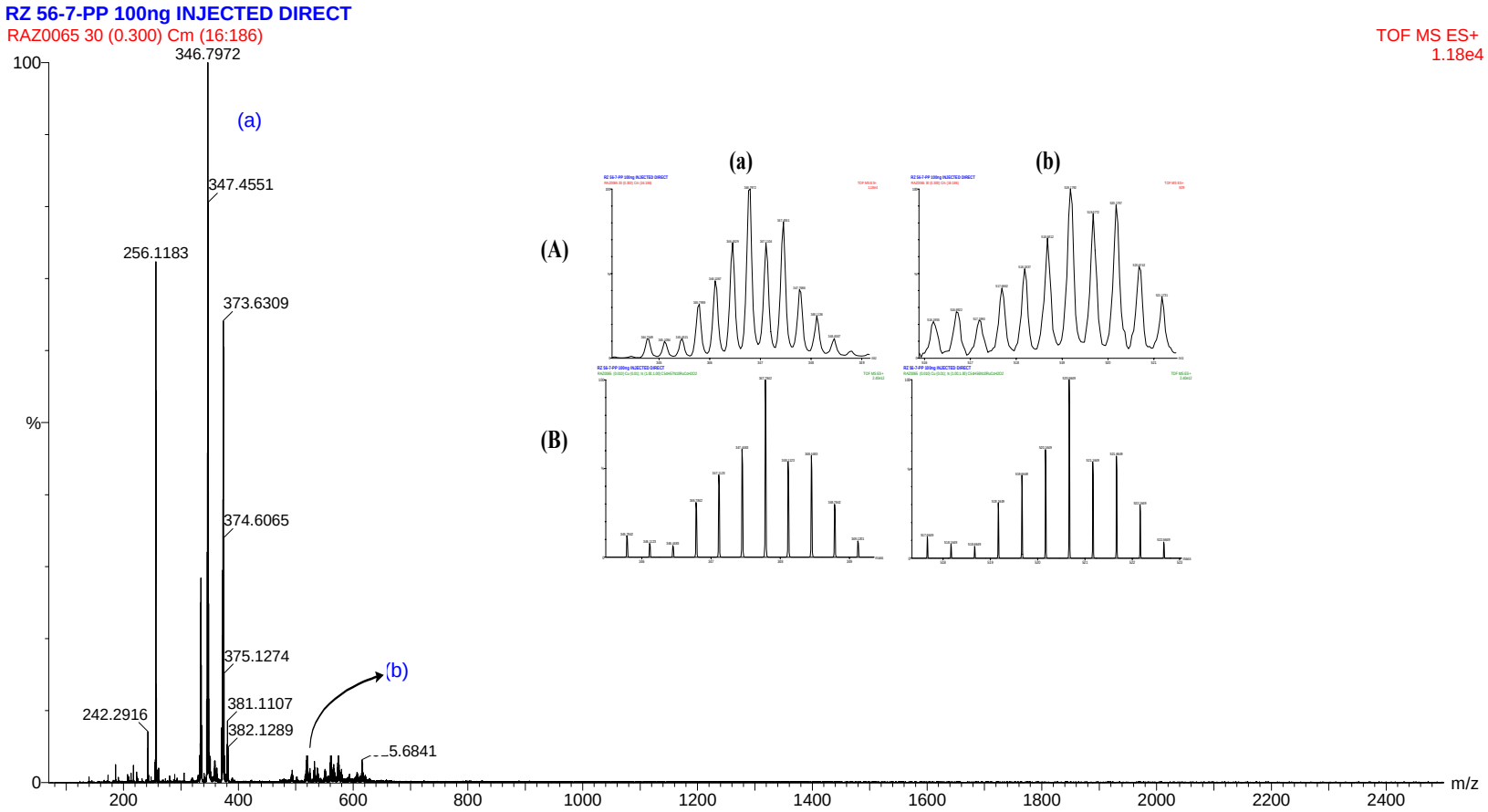


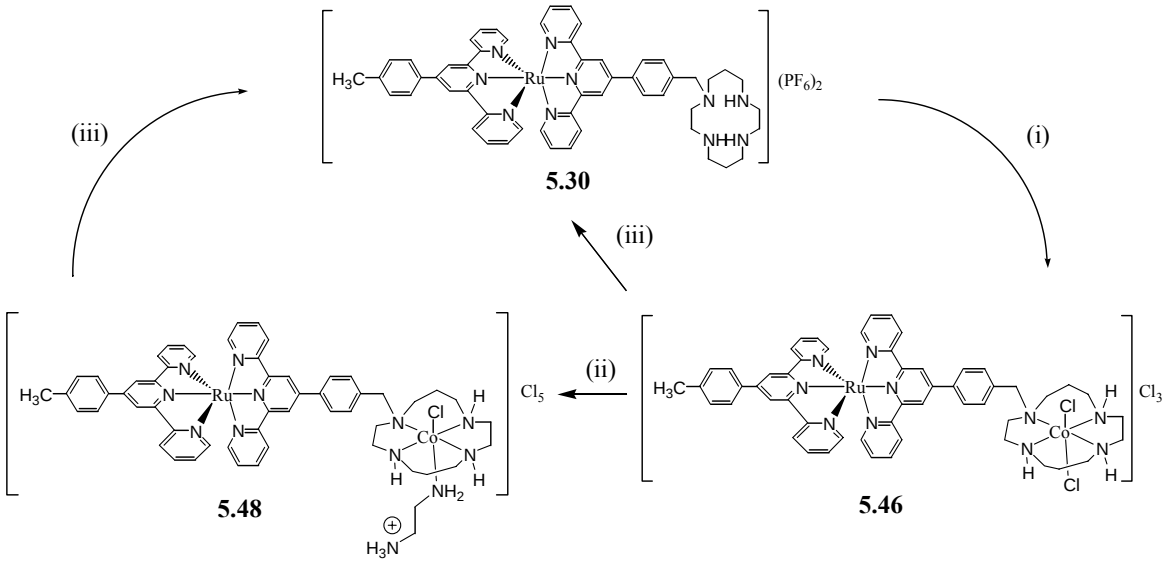
Figure 5.9. ESI-MS of $[(ttp)Ru(cymt)Co(OH)_2](PF_6)_3$ complex, **5.45**, in CH_3CN. (A) High resolution scans of the two major signals (a), and (b); (B) Calculated isotope distribution patterns for (a) $[(ttp)Ru(cymt)Co(OH)_2]^{3+}$ and (b) $[(ttp)Ru(cymt)Co(OH)_2+(PF_6)]^{2+}$, at *m/z* 346.80 and 519.18, respectively.

From the viewpoint of HSCs development, a more relevant model for an acceptor subunit based on cyclam ligand in our systems would contain a bidentate amine coordinated to the Co(III) in *cis* geometry. The bidentate ligand en was chosen; because it is readily available and also it is a close structural analogue for some nitrogen mustards (see Chapter 1). A good model compound for the cobalt end of the target molecule exists as the crystal structure of *cis*-(1,4,8,11-tetraazacyclotetradecane) ethylenediaminecobalt(III) chloride trihydrate complex has been reported in the literature.[486]

During our research, attempts to synthesise the Ru(II)-Co(III) complex **5.47** (Scheme 5.10) have been made under a variety of conditions. When an excess of the en ligand in MeOH was allowed, by warming, to react with complex **5.46**, the parent complex **5.30** in which the cobalt ion has been removed from the macrocycle was regenerated in 98% yield. This result was also confirmed using ^{1}H NMR, ^{13}C NMR, and ESI-MS techniques. However, similar results were observed when milder reaction conditions (for example, 2:1 molar ratio of en/complex at 30ºC) was applied. ESI-MS results for both sets of reactions in MeOH solutions reveal large signals that can be assigned to $[(ttp)Ru(cymt)+H]^{3+}$ cationic species (see Figure 5.5).

More interestingly, the reaction in which an equimolar ratio of en at 30ºC in MeOH was used, did not undergo complete removal of the Co(III) ion from the complex. ^{1}H NMR and ^{13}C NMR spectra of the product exhibit peaks which can be attributed to the Ru(II) complex **5.30** as well as other species associated with dinuclear Ru(II)-Co(III) starting material. ESI-MS of the product in MeOH solution shows a prominent ion at a mass-to-charge ratio (*m/z)* of 315.7538, with mass and isotopic distribution pattern corresponding to $[(ttp)Ru(cymt)+H]^{3+}$ cation along with other signals at 392.1049, and 605.1373 that can be assigned to

$[(ttp)Ru(cymt)Co(en)Cl_3+H]^{3+}$, and $[(ttp)Ru(cymt)Co(en)Cl_4+H]^{2+}$, respectively. In the last two species, the en ligand may be acting as a monodentate ligand *trans* to the coordinated Cl^- ion in the complexes. This would allow the other end of the en ligand to be protonated. Protonation of the en ligand would presumably inhibit its ability to act as a bidentate ligand in the complexes (Scheme 5.11).



Scheme 5.11. Proposed mechanism of addition reaction of en with the dinuclear complex **5.46**. (i) 1. $Na_3[Co(NO_2)_6]$, acetone/water (1:2), 50 ºC, overnight, Et_3N or NaOH; 2. 6M HCl, steam bath, 8 hr; (ii) en (equimolar), MeOH, 30ºC (iii) excess en, MeOH, 30ºC.

Funston's attempts to prepare cyclam complexes containing primary and secondary amines *via* amine ligand exchange with $[Co(cyclam)Cl_2]Cl$, or beginning from cobalt(II) salts, cyclam, and an amine were also unsuccessful and they also failed to isolate solid products containing these amines.[42]

An alternative approach to overcome this problem would be using the Ru(II) complex **5.43** containing cyclen (Scheme 5.9) instead to react with Co(III) ions. It is

well established in the literature[489-492] that in contrast to cyclam which is coordinated in a planar structure, the cavity formed by cyclen is too small to accommodate a transition metal ion (such as Co(III)) in the plane of the N(4) donor atoms. Cyclen is usually coordinated in a folded *cis*- arrangement. (Figure 5.10)

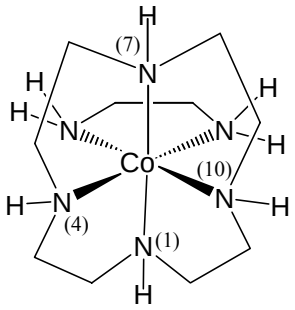


Figure 5.10. Representation of a $[Co(cyclen)(en)]^{3+}$ ion showing a *cis*-folded geometry. Isomers are not shown.[491]

As was mentioned in Chapter 1, we were interested in reducing the number of stereoisomers that may produced upon coordination of the mono-N-functionalised macrocycle to metal atoms. We thought that N-functionalisation of the macrocycles using bisaminal strategy might be more useful.

Ligands tcnt, **1.76**, and pcymt, **5.35**, may well fit into this strategy. Ligand tcnt, **1.76**, that has both a meridionally coordinating subunit (tpy), and a facially coordinating triazamacrocycle (Figure 5.2) has been synthesised using the approach explained above (Scheme 5.4) which gives only one isomer when the tpy is introduced to a $Ru(tpy)Cl_3$ precursor and the azamacrocycle coordinates to another octahedral metal centre.

Unfortunately, reactions of Ru(II) complex **5.40** (containing tcnt) containing ligand tcnt, **1.76**, with $Na_3[Co(NO_2)_6]$, $[Co(tacn)Cl_3]$, $[Co(dien)Cl_3]$, or $[Co(dien)(H_2O)_3]$ under a variety of conditions proved unsuccessful. We have also found that when Ru(II) complex **5.40** was mixed with $[Co(tacn)Cl_3]$ in aqueous

solutions, [Co(tacn)Cl_3] reacted with itself rather than with the Ru(II) complex **5.40**. This reaction produces oxo-bridged Co(III) complexes. For example, during one of the attempts, bis(μ-oxo)-(μ-nitrato-O,O')-bis((1,4,7-triazanonane)-cobalt(III) chloride complex (Figure 5.11) was formed when Ru(II) complex **5.40** was treated with [Co(tacn)Cl_3] in aqueous acetone solution, followed by column chromatography. Unfortunately, while the structural solution could be found, the refinements for the lattice water molecules were unsatisfactory ($R_1 \approx 5\%$).

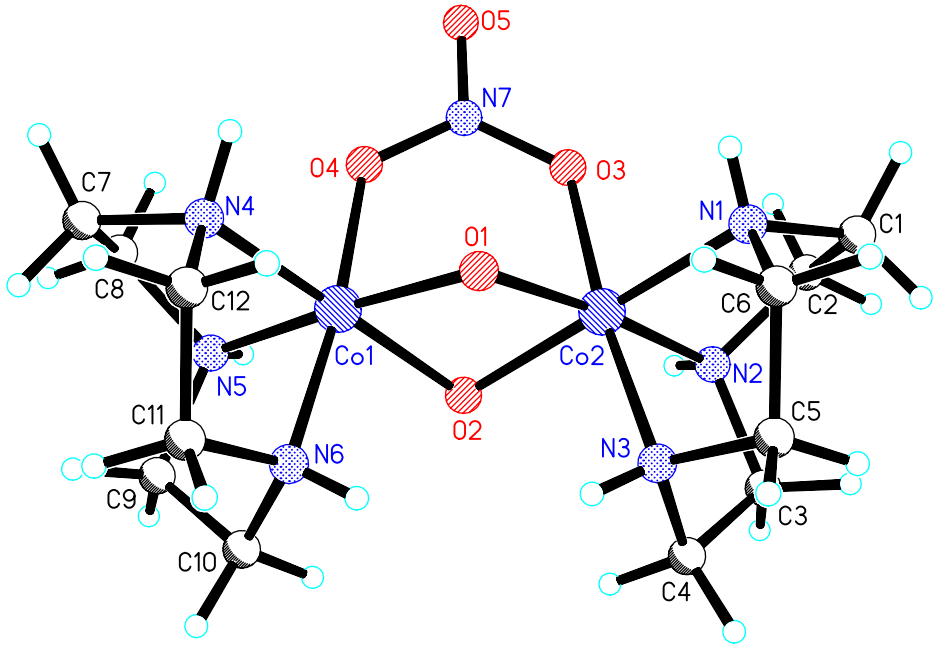


Figure 5.11. X-ray crystal structure of bis(μ-oxo)-(μ-nitrato-O,O')-bis((1,4,7-triazanonane)-cobalt(III) chloride complex.

There is no evidence of any coordination to the tacn site, when Ru(II) complex **5.40** was treated with $CuCl_2$, $Cu(NO_3)_2$, $Cu(ClO_4)_2$, $Ni(ClO_4)_2$, $FeCl_2$, $Fe(ClO_4)$, $ZnCl_2$, $Zn(NO_3)_2$, $AgBF_4$, $Ag(ClO_4)$, or $Cd(NO_3)_2$ under a variety of conditions. In

some cases, precipitates were formed, but these proved to be the complex **5.40** with different anions.

Reactions of Ru(II) complex **5.37** (containing dint) with $Na_3[Co(NO_2)_6]$, $[Co(dien)Cl_3]$, or $[Co(dien)(H_2O)_3]$ under a variety of conditions afforded products whose ^{1}H NMR signals were broad. ESI-MS also indicated coordination of Co(III) ion at the amine site. Reactions of Ru(II) complex **5.37** with $CuCl_2$, $Cu(NO_3)_2$, $Cu(ClO_4)_2$, $Ni(ClO_4)_2$, $FeCl_2$, $Fe(ClO_4)$, $ZnCl_2$, $Zn(NO_3)_2$, $AgBF_4$, or $Ag(ClO_4)$, were also attempted. We found that Ru(II) complex **5.37** reacted with $Ni(ClO_4)_2$, $AgBF_4$, and $Ag(ClO_4)$ at its dien site under our conditions. Figure 5.12 shows selected signals in the ESI-MS of the reaction products of Ru(II) complex **5.37** with $Ni(ClO_4)_2$, $Ag(ClO_4)$, and $Na_3[Co(NO_2)_6]$ in different reactions. Signals (a), (b), (c), (d), (e), and (f) correspond to $[(ttp)Ru(dint)Ni(ClO_4)(PF_6)]^{2+}$, $[(ttp)Ru(dint)Ni(ClO_4)_2]^{2+}$, $[(ttp)Ru(dint)Ni(PF_6)]^{3+}$, $[(ttp)Ru(dint)Ag(ClO_4)]^{2+}$, $[(ttp)Ru(dint)Co(NO_2)_3(PF_6)]^{+}$, and $[(ttp)Ru(dint)Co(NO_2)_3]^{2+}$ cations at *m/z* 577.5750, 554.5671, 351.0659, 529.5778, 1191.2150, and 523.1514, respectively. The observed isotope patterns are a close match to the calculated isotopic distribution patterns for ions of those compositions. In all of the spectra, however, major signals corresponding to the parent Ru(II) complex **5.37** were also observed.

Figure 5.13 represents the ^{1}H NMR spectra of the Ru (II) complex **5.37** and its reaction product with $AgClO_4$ in dmso-d_6 solutions showing significant changes in the chemical shifts associated with protons signals of the amine side chains in the complex. The chemical shifts of the protons in the aromatic region, however, remained unchanged. ESI-MS and NMR observations suggest coordination of silver ions at the amine site of the complex upon reaction with $AgClO_4$. Similar results were observed when $AgBF_4$ was used.

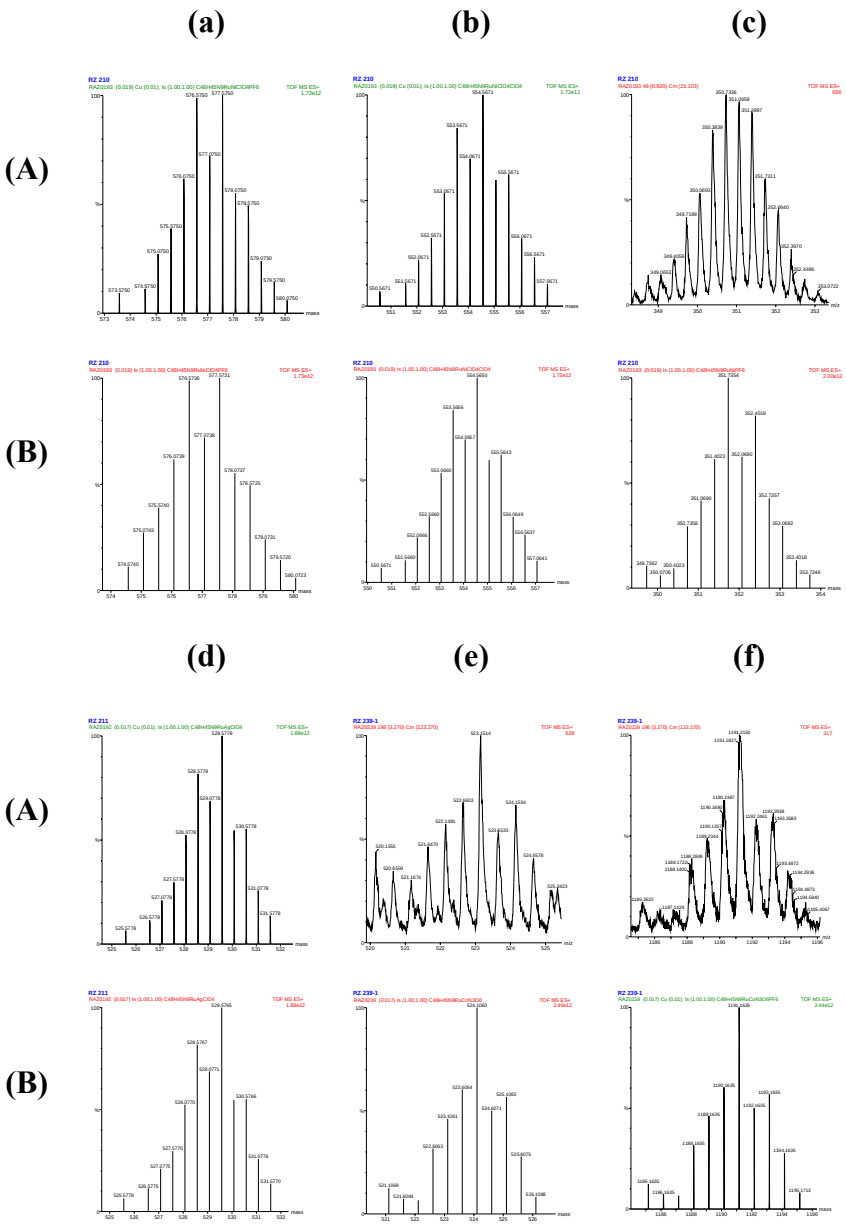


Figure 5.12. ESI-MS of selected signals of the reaction products of Ru(II) complex **5.37** with $Ni(ClO_4)_2$ and $Ag(ClO_4)$ in different reactions. **(A)** High resolution scans of the selected signals**(a)**, **(b)**, **(c)**, **(d)**, **(e)**, and **(f)**; **(B)** Calculated isotopic distribution patterns for **(a)** $[(ttp)Ru(dint)Ni(ClO_4)(PF_6)]^{2+}$, **(b)** $[(ttp)Ru(dint)Ni(ClO_4)_2]^{2+}$, **(c)** $[(ttp)Ru(dint)Ni(PF_6)]^{3+}$, **(d)** $[(ttp)Ru(dint)Ag(ClO_4)]^{2+}$, **(e)** $[(ttp)Ru(dint)Co(NO_2)_3(PF_6)]^{+}$, and **(f)** $[(ttp)Ru(dint)Co(NO_2)_3]^{2+}$, at m/z 577.5750, 554.5671, 351.0659, 529.5778, 1191.2150, and 523.1514, respectively.

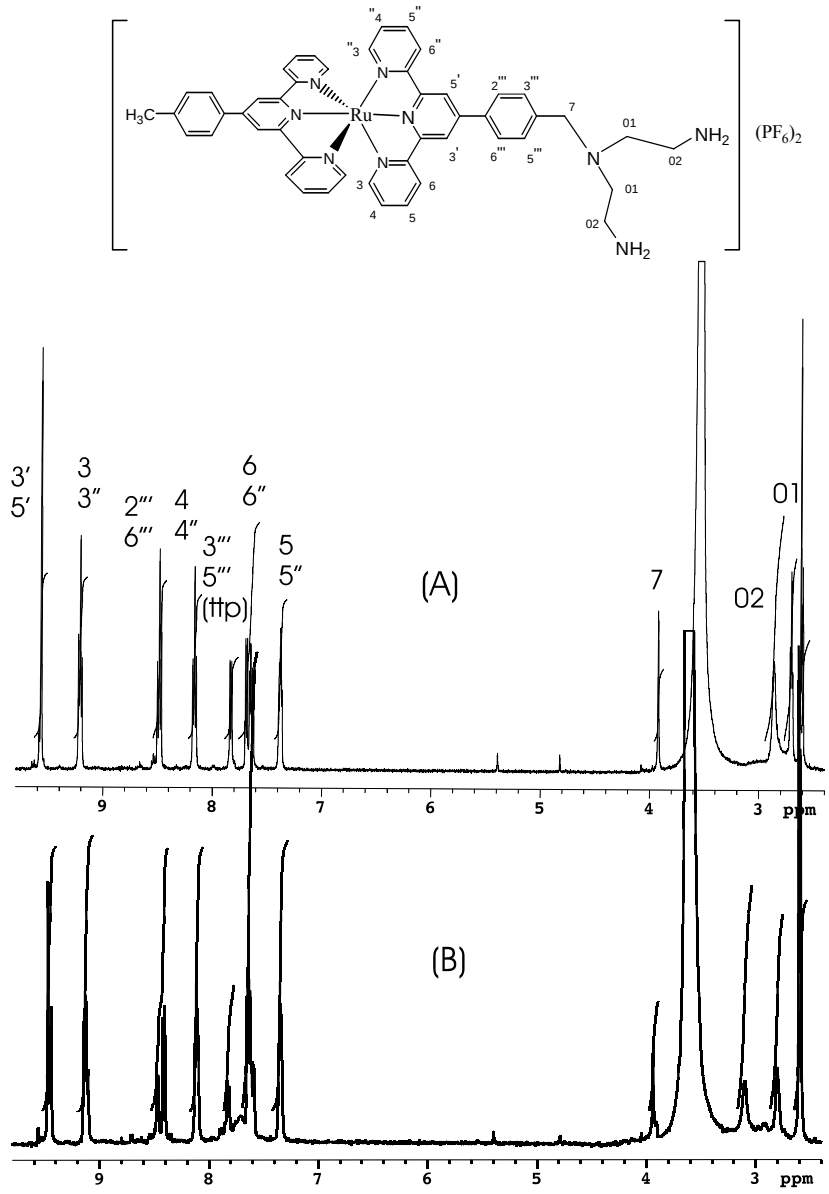


Figure 5.13. ^{1}H NMR spectra of the Ru (II) complex **5.37** (containing dint), **(A)**, and its reaction product with $AgClO_4$, **(B)**, in dmso-d_6 solutions. The chemical shifts associated with the protons H_{01} and H_{02} changed significantly when complex **5.37** was allowed to react with $AgClO_4$ in acetone.

There is no evidence of any coordination to the dien site of the complex **5.37** with the other transition metal ions that we studied.

As was also mentioned above, ring cleavage reduction of Ru(II) complex **5.41** (Scheme 5.9) resulted in a low yield of a new complex, **5.42**, in which a piperazinocyclam ring is appended to the terpyridine unit. Unfortunately, attempts to purify the crude product have proved unsuccessful, and further coordination chemistry of **5.42** could not be attempted. Optimising the yield of the reaction and also its purification may be a subject of the future studies.

5.5. Conclusion

Three new ditopic ligands bpat, **5.31**, dint, **5.33**, and cynt, **1.75**, based on tridentate planar terpyridyl systems (Scheme 5.8) have been synthesised using direct/indirect alkylation methodologies in relatively high yields. Our attempts to improve the yield of the reaction/s to synthesise the new ligand pcymt, **5.35**, using bisaminal methodology, proved to be unsuccessful.

Seven new Ru(II) complexes, **5.37** (containing dint), **5.31** (containing bpat), **5.39, 5.40** (containing tcnt), **5.41**, **5.42** (containing pcymt), and **5.43** (containing cynt)**,** were prepared using either the unprotected ligands or the ligands precursors (Scheme 5.9). Our attempts to increase the yield of the reductive ring cleavage reaction of **5.41** have so far proved unsuccessful.

Reactions of the Ru(II) complexes **5.30** (containing cymt), **5.37** (containing dint), **5.31** (containing bpat), and **5.40** (containing tcnt) (Scheme 5.9) with a range of transition metal ions including Co(III) ion were investigated. We found that the complexes **5.30** and **5.37** containing cycalm and dien subunits, respectively, are most likely to react with Co(III) ion. We also showed that **5.37** reacts with $Ni(ClO_4)_2$, and sliver salts.

In this Chapter, we focused on the synthesis and characterisation of some heterodinuclear Ru(II)-Co(III) complexes using the Ru(II) complex **5.30** as a starting material. Unfortunately, attempts to synthesise the heterodinuclear complex **5.47** (Scheme 5.10) failed under our conditions. The reason why the Ru(II)-Co(III) complex **5.46** (Scheme 5.10) in the presence of en ligand undergoes decomposition is not understood at this time; but it may be related to the high charge on the complex.

Some evidence for the importance of charge in the chemistry of these systems can be found in the isolation of **5.45**. In this complex, the Co(III) ion is coordinated by a cyclam unit and two hydroxide ligands. The related Co(III)-cyclam complex[486] has been isolated with two water molecules coordinated to Co(III) ion. Presumably the higher charge (due to the presence of Ru(II) in the complex) leads to a greater tendency for these water ligands to be deprotonated, forming the hydroxido complex.

Mild reaction conditions appear promising, in that some evidence for the formation of heterodinuclear complexes with en bound to Co(III) ion has been found. This is an obvious avenue for further study. For example, reactions using mono-N-substituted cross-bridged cyclams will be useful. The cyclam binding sites in these systems provide *cis*-arrangements suitable for binding bidentate en ligands to Co(III) ions. A proposed synthetic strategy to prepare mono-N-substituted cross-bridged cyclam will be discussed later in Chapter 7.

Ru(II) complex **5.43** containing a cyclen macrocyle is another potential candidate for preparation of heterodinuclear Ru(II)-Co(III) complexes as HSC model systems in future. The azamacrocycle cyclen provides a *cis*-folded geometry upon coordination with Co(III) which is suitable for binding a bidentate en ligand. A proposed synthetic methodology based on bisaminal approach using cyclam/cyclen will also be presented in Chapter 7 as potential work for the future.

Chapter 6

Miscellaneous Chemical Structures

6.1. Introduction

As was explained in Chapter 1, ditopic ligands 2,4-bis(2'-pyridyl)-6(4"-pyridyl)-1,3,5-triazine (bppt), **2.1**, and 2,4,6-tris(pyridyl)-1,3,5-triazine (tpt), **2.6**, are potentially suitable candidates for preparing heterodinuclear systems. Ligands **2.1** and **2.6** contain a tridentate binding site and a monodentate or bidentate binding subunits, respectively.

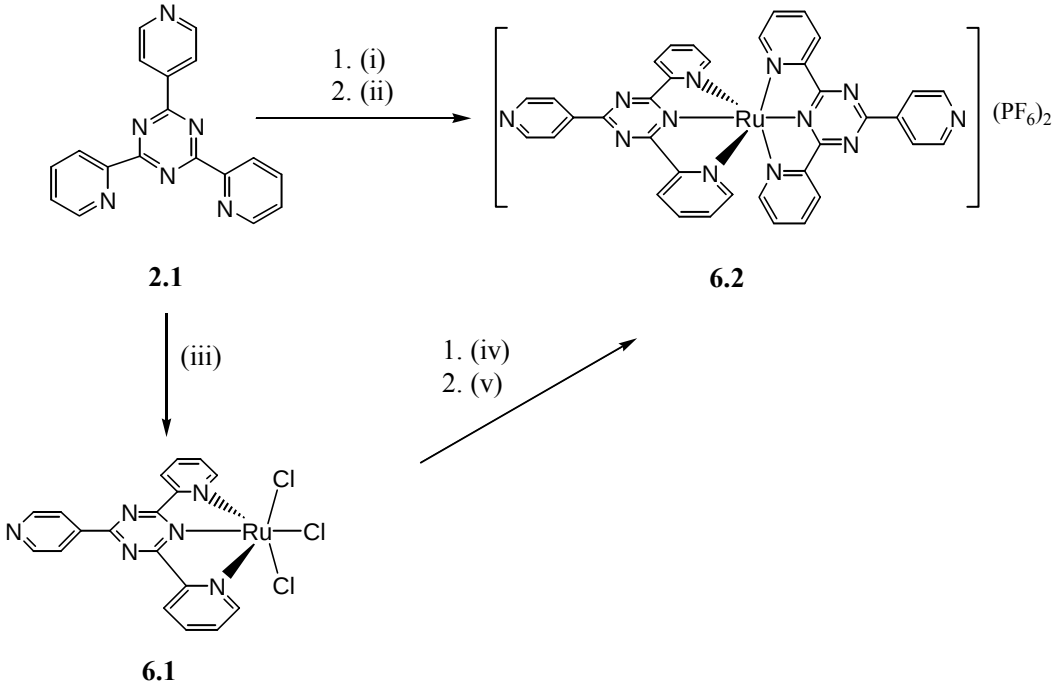
In Chapter 2, synthesis and characterisation of some Ni(II) complexes of tpt ligand was explained. We were, initially, interested in using complex precursor $Ru(ttp)Cl_3$, or Ru(II) and iron(II) salts for binding to the tridentate sites in these ligands, selectively. The remaining monodentate or bidentate sites in **2.1** and **2.6**, respectively, were considered as suitable binding sites for different ions (such as Co(III) ion in our study). This chapter describes the synthesis of some ruthenium and iron complexes of bppt and tpt ligands, respectively. X-ray crystal structures of two ruthenium and iron complexes are also explained.

During my research project, some complexes were also incidentally prepared and crystallographically characterised. This Chapter explains, for example, the

structure of a copper complex which was obtained during the reaction of the ditopic ligand cymt**, 1.74,** (in Chapter 5) with $Cu(NO_3)_2$ salt.

6.2. Ru Complexes of 2,4-bis(2'-pyridyl)-6(4"-pyridyl)-1,3,5-triazine Ligand

Ruthenium complex **6.2** (Scheme 6.1) was synthesised in two routes. Two-step reaction of the 2,4-bis(2'-pyridyl)-6(4"-pyridyl)-1,3,5-triazine ligand (bppt), **2.1**, with $RuCl_3.3H_2O$ followed by equimolar addition of the ligand proved to be more efficient than one-step reaction of $RuCl_3.3H_2O$ with the ligand (12% *vs* 6%). In each case, the product was isolated as its PF_6^- salt. ^1H NMR, ^{13}C NMR, and ESI-MS data for the complex were all consistent with the structure.



Scheme 6.1. Synthesis of Ru complexes of ligand bppt, **2.1**. (i) $RuCl_3.3H_2O$, ethylene glycol, reflux, 15min; (ii) NH_4PF_6; (iii) $RuCl_3.3H_2O$, EtOH, reflux, 4 h; (iv) $RuCl_3.3H_2O$, **2.1** (equimolar), ethylene glycol, reflux, 15min; (v) NH_4PF_6.

6.2.1. Structure of $[Ru(bppt)_2](PF_6)_2$ Complex

metal-organic papers

Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis[2,4-bis(2-pyridyl-κN)-6-(4-pyridyl)-1,3,5-triazine-κN^3]ruthenium(II) bis(hexafluorophosphate)

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Received 15 April 2005
Accepted 20 April 2005
Online 27 April 2005

Key indicators

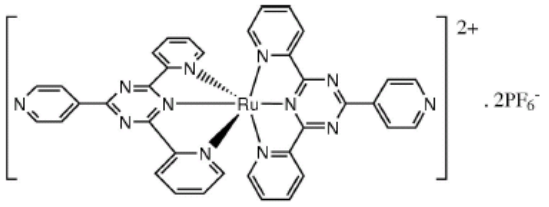
Single-crystal X-ray study
T = 85 K
Mean σ(C–C) = 0.008 Å
R factor = 0.058
wR factor = 0.137
Data-to-parameter ratio = 13.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The coordination of Ru in the title compound, $[Ru(C_{18}H_{12}N_6)_2](PF_6)_2$, is octahedral. The coordination sphere is formed by six N atoms from the tridentate sites of two ligands that are meridionally oriented around the Ru^{II} ion. The locations of the donor atoms deviate substantially from an ideal octahedron. The Ru—N distances are in the range 1.983 (4)–2.111 (4) Å. The Ru atom occupies a special position with twofold rotational symmetry. One of the P atoms occupies a special position with twofold rotational symmetry and the other P atom is located on a special position at a centre of symmetry.

Comment

Our aim involves the synthesis and characterization of bridging ligands containing two non-equivalent binding sites. Our interests are particularly focused on preparation of such ligands capable of binding an Ru^{II} ion at one site and a Co^{III} ion at the other site. As part of our study, the ligand 2,4-bis(2-pyridyl)-6-(4-pyridyl)-1,3,5-triazine was synthesized using the method of Polson *et al.* (2002). This terpyridyl-like ligand, which contains a remote fourth N-donor atom, was a candidate for our study. The title ruthenium complex, (I), in which two ligands are coordinated to an Ru^{II} ion through their tridentate sites was prepared according to the reported methods (Polson *et al.*, 2002, 2004).



(I)

Complex (I) (Fig. 1) crystallizes in the orthorhombic space group *Pcca* with four well separated molecules in the unit cell. The asymmetric unit comprises one half-cation and two half-anions. The Ru atom occupies a special position with twofold rotational symmetry. One of the P atoms occupies a special position with twofold rotational symmetry and the other P atom is located on a special position at a centre of symmetry. The ruthenium ion is six-coordinate, but the geometry is significantly distorted from that of an ideal octahedron as a result of the constraints enforced by the ligand structure. This is commonly observed for terpyridyl-like ligands (Thummel &

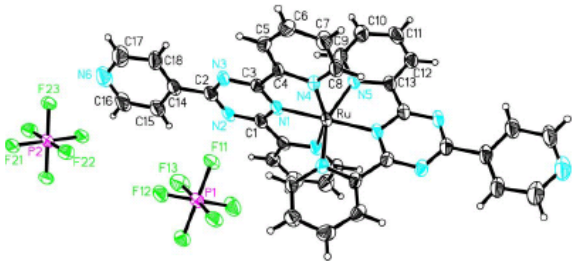


Figure 1
A perspective view of the Ru^{II} complex (I), showing the atom-labelling scheme with 50% probability displacement ellipsoids. Unlabelled atoms in the cation and PF_6^- anions P_1 and P_2 are related by the symmetry codes $(\frac{1}{2}-x, -y, z)$, $(\frac{1}{2}-x, 1-y, z)$ and $(1-x, 2-y, -z)$, respectively. H atoms are drawn as small spheres of arbitrary radii.

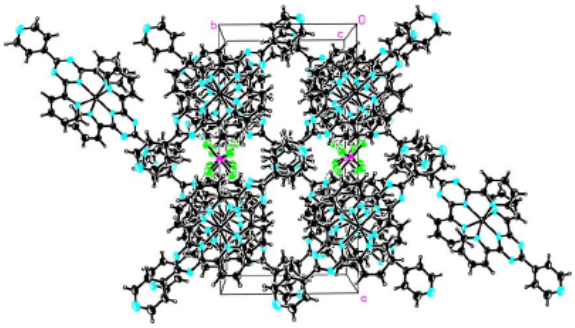


Figure 2
A packing diagram, showing the π–π stacking interactions between the complexes parallel to the *ac* plane.

Jahng, 1986; Constable *et al.*, 1990, 1992, 2001; Cathey *et al.*, 1990; Bushell *et al.*, 1998; Craig *et al.*, 1998; Sasaki *et al.*, 1998; Cardenas *et al.*, 1999; Chamchoumis & Potvin, 1999; Fallahpour *et al.*, 1999; Lashgari *et al.*, 1999; Pyo *et al.*, 1999; Ziegler *et al.*, 1999; Alcock *et al.*, 2000; Encinas *et al.*, 2002; Fang *et al.*, 2002; Laine *et al.*, 2002). Each of the tridentate ligands occupies three meridionally located coordination sites. The Ru—N bond lengths show the expected pattern for complexes related to $[Ru(terpy)_2](PF_6)_2$ (Pyo *et al.*, 1999; Lashgari *et al.*, 1999), with short metal–ligand bonds to the central ring [1.983 (4) Å] and longer bonds to the side pyridine rings [mean 2.108 (4) Å]. The non-coordinated pyridine rings are not coplanar with the central triazine ring and form interannular angles of 16.8 (3)°. Similar observations have been made for the Ni^{II} and Ru^{II} complexes based on the 4′-phenyl-substituted terpyridine system (Constable *et al.*, 1990; Chamchoumis *et al.*, 1999). As shown in Fig. 2, there are π–π stacking interactions (face–face) between the terminal pyridyl rings of adjacent complexes [centroid–centroid distance = 3.72 (8) Å]. The complex cations are arranged like the steps of a spiral staircase along the *b* axis of the unit cell, with the stack of pyridyl rings forming the pole of the staircase. There appears to be no significant hydrogen bonding in the crystal structure.

Experimental

The 2,4-bis(2-pyridyl)-6-(4-pyridyl)-1,3,5-triazine ligand and its ruthenium complex, (I), were synthesized according to the methods of Polson *et al.* (2002, 2004). Red block-shaped crystals were obtained by slow evaporation of a CH_3CN–H_2O solution of the complex (yield 16%).

Crystal data

$[Ru(C_{18}H_{12}N_6)_2](PF_6)_2$
$M_r = 1015.68$
Orthorhombic, *Pcca*
$a = 21.474$ (11) Å
$b = 11.008$ (6) Å
$c = 16.044$ (8) Å
$V = 3793$ (3) Å^3
$Z = 4$
$D_x = 1.779$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8243 reflections
$\theta = 4.9$–$52.5°$
$\mu = 0.61$ mm^{-1}
$T = 85$ (2) K
Block, red
0.61 × 0.29 × 0.08 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.709$, $T_{max} = 0.953$
21 050 measured reflections
3864 independent reflections
2935 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 26.4°$
$h = -26 \rightarrow 26$
$k = -13 \rightarrow 11$
$l = -18 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.137$
$S = 1.16$
3864 reflections
287 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0303P)^2 + 19.9599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.54$ e Å^{-3}
$\Delta\rho_{min} = -1.09$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ru—N1	1.983 (4)	Ru—N5	2.111 (4)
Ru—N4	2.104 (4)		
$N1^i$—Ru—N1	178.9 (3)	N1—Ru—$N5^i$	77.41 (18)
$N1^i$—Ru—$N4^i$	77.00 (17)	$N4^i$—Ru—$N5^i$	92.97 (17)
N1—Ru—$N4^i$	102.22 (16)	N4—Ru—$N5^i$	154.38 (16)
N1—Ru—N4	77.00 (17)	$N1^i$—Ru—N5	77.41 (18)
$N4^i$—Ru—N4	93.6 (2)	$N5^i$—Ru—N5	91.7 (2)
$N1^i$—Ru—$N5^i$	103.38 (17)		
N3—C2—C14—C18	16.9 (8)	N2—C2—C14—C15	16.2 (8)

Symmetry code: (i) $\frac{1}{2}-x, -y, z$.

All H atoms were placed in calculated positions [C—H = 0.93 Å and $U_{iso} = 1.2U_{eq}(C)$] and were included in the refinement in the riding-model approximation. The highest peak is very close to Ru (0.89 Å) and the deepest hole is near N4 (1.62 Å), neither in chemically sensible locations for atoms.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

RZ would like to acknowledge receipt of a University of Canterbury doctoral scholarship.

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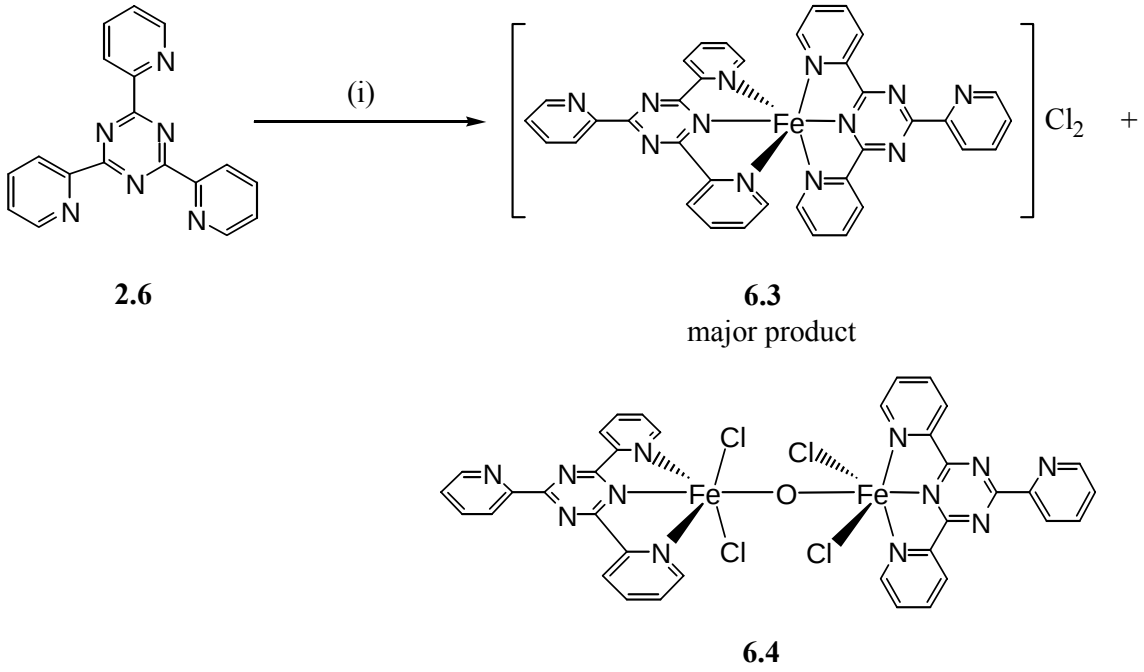
6.3. Iron Complexes of 2,4,6-Tris(2-pyridyl)-1,3,5-triazine Ligand

A deep-purple solution was obtained when a methanolic solution of $FeCl_2.4H_2O$ was added to a solution of the ligand tpt, **2.6**, in MeOH (Scheme 6.2). Slow evaporation of the mixture at room temperature afforded two different crystals successively: dark navy-blue prisms of **6.3** as the major product were formed first, followed by formation of orange rod-like crystals of **6.4**.



2.6

6.3
major product

6.4

Scheme 6.2. Synthesis of iron complexes of tpt ligand. (i) $FeCl_2.4H_2O$, MeOH.

X-ray diffraction of dark navy-blue crystals revealed that the iron complex **6.3** was formed (Scheme 6.2), but the marginal quality of the crystals meant that while a structural solution could be found, the refinement was unsatisfactory ($R_1 \approx 15\%$). The tentative structure, shown in Scheme 6.2, is that of a complex, with iron bound in the terpyridyl sites of two tpt ligands, leaving the bidentate site of the ligand uncoordinated. ^{1}H NMR, ^{13}NMR, ESI-MS, and elemental analysis of the complex are all consistent with the proposed structure.

In this section, the X-ray crystal structure of the minor product, **6.4**, (Scheme 6.2) has been explained.[493]

6.3.1. Crystal Structure of The Iron Complex 6.4

The complex $[Fe(ttp)(Cl)_2]_2(\mu\text{-}O).2H_2O$, **6.4**, was crystallized in the monoclinic space group $P2_1/c$. As shown in Figure 6.3, each Fe ion is coordinated to tridentate terpyridyl site of the ligand tpt, **1.70**, in the complex. The fourth and the fifth coordination sites of each Fe ion are also occupied by two Cl ions *cis* to the oxo bridging ligand. The tpt ligands act as planar tridentate binding moiety while the remaining bidentate binding sites in the ligands remain non-coordinated. Bond angles show that coordination at each Fe centre deviates from octahedral symmetry (see caption of Figure **6.3**).

The Fe-O-Fe angle (176.12(15) º) is close to those observed for the similar oxo bridged polypyridyl complexes of Fe(III) (Table 6.1) and is marginally different to the other reported structures (Table 6.2). In the structures where Fe-O-Fe angles are 180º,[494-499] the bridged O atoms are on the special positions and the Fe-O-Fe angles are constrained at 180º by the crystallographically imposed centrosymmetry.

Table 6.1. Literature Fe-O- Fe bond angles close to structure **6.4**.

Structure	Fe-O-Fe angle (º)
$C_{36}H_{48}Cl_2Fe_2N_8O_3,2(ClO_4)$[500]	176.0(5)
$C_{32}H_{44}Cl_2Fe_2N_8O,2(ClO_4),0.751(H_2O)$[501]	176.42
$C_{32}H_{44}Cl_2Fe_2N_8O,2Cl,C_2H_3N,5(H_2O)$[502]	177.76
$C_{44}H_{38}Cl_2Fe_2N_{10}O,2Cl,1.5(CH_4O)$[503]	178.6(4)

Table 6.2. Literature Fe-O- Fe bond angles different from structure **6.4**.

Structure	Fe-O-Fe angle (º)
$C_{48}H_{32}Cl_2Fe_2N_8O,2Cl,4.5(H_2O)$[504]	161(1)
$C_{48}H_{48}Cl_2Fe_2N_8O,2(F_6P)$[505]	167.4(4)
$C_{40}H_{32}Cl_2Fe_2N_8O,2(ClO_4),0.25(C_2H_3N),0.25(CH_4O),0.25(H_2O)$[506]	166.91
C36H36Cl2Fe2N8O,2(ClO4)[507]	174.7(5)
$C_{36}H_{38}Cl_2Fe_2N_{10}O_3,2(NO_3)$[494]	180.0
$C_{48}H_{42}Cl_2Fe_2N_{14}O,2(ClO_4),5(CH_4O),2(H_2O)$[495]	180.0
$C_{40}H_{28}Cl_2Fe_2N_8O,2(ClO_4),2(H_2O)$[496]	180.0
$C_{28}H_{32}Cl_2Fe_2N_8O_3,2(ClO_4),2(H_2O)$[500]	180.0
$C_{34}H_{38}Cl_2Fe_2N_{10}O,2Cl,4(CH_4O)$[499]	180.0
$2(C_{18}H_{18}Cl_2FeN_4),C_{36}H_{36}Cl_2Fe_2N_8O,4(ClO_4)$[497]	180.0
$C_{48}H_{42}Cl_2Fe_2N_{14}O,2(F_6P),2(C_4H_8O)$[498]	180.0

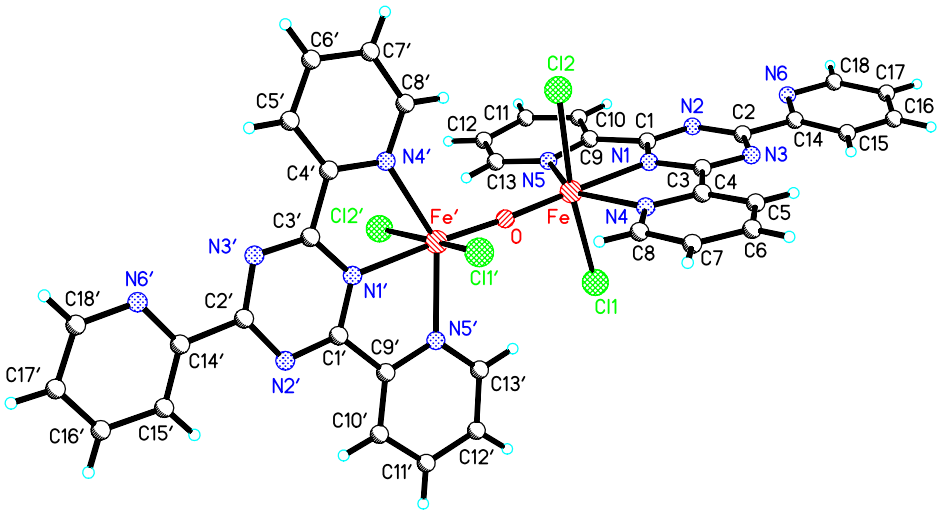


Figure 6.3. A diagram of the diiron oxo-bridged complex **6.4** with two solvated water molecules are omitted for clarity. Selected bond lengths [Å] and angles [°]:Fe-O 1.767(2); Fe-N(1) 2.124(2); Fe-N(5) 2.186(3); Fe-N(4) 2.202(3); Fe-Cl(2) 2.3999(9); Fe-Cl(1) 2.4187(9); Fe'-O 1.768(2); Fe'-N(1') 2.127(3); Fe'-N(4') 2.189(3); Fe'-N(5') 2.193(3); Fe'-Cl(1') 2.4051(10); Fe'-Cl(2') 2.4262(10); O-Fe-N(1) 178.67(10); O-Fe-N(5) 108.07(10); N(1)-Fe-N(5) 72.92(10); O-Fe-N(4) 105.96(10); N(1)-Fe-N(4) 73.03(10); N(5)-Fe-N(4) 145.95(10); O-Fe-Cl(2) 94.97(8); N(1)-Fe-Cl(2) 85.96(7); N(5)-Fe-Cl(2) 86.49(7); N(4)-Fe-Cl(2) 91.71(7); O-Fe-Cl(1) 95.12(8); N(1)-Fe-Cl(1) 84.01(7); N(5)-Fe-Cl(1) 86.78(7); N(4)-Fe-Cl(1) 89.16(7); Cl(2)-Fe-Cl(1) 169.21(3); O-Fe'-N(1') 177.05(11); O-Fe'-N(4') 109.86(10); N(1')-Fe'-N(4') 73.07(10); O-Fe'-N(5') 104.16(10); N(1')-Fe'-N(5') 72.91(10); N(4')-Fe'-N(5') 145.95(10); O-Fe'-Cl(1') 95.00(8); N(1')-Fe'-Cl(1') 84.87(8); N(4')-Fe'-Cl(1') 86.72(8); N(5')-Fe'-Cl(1') 91.64(8); O-Fe'-Cl(2') 95.03(8); N(1')-Fe'-Cl(2') 85.37(8); N(4')-Fe'-Cl(2') 85.08(8); N(5')-Fe'-Cl(2') 90.85(8); Cl(1')-Fe'-Cl(2') 168.74(4); Fe-O-Fe' 176.12(15).

In the structure **6.4**, the Fe-O bond lengths are at 1.767(2) and 1.768(2) Å; whereas this distance in the literature structures are at 1.782(1),[503] 1.811(1),[501] 1.603(1),[502] 1.780(2),[500] 1.800(8),[500] 1.794(8),[500] 1.787(6),[504] 1.786(2),[505] 1.788(4),[506] 1.785(1),[507] 1.7816(7),[494] 1.80,[495] 1.779(1),[496] 1.788(4),[499] 1.790(1),[497] and 1.8050(9) Å.[498]

Moreover, the Fe-Cl bond distances in the structure **6.4** are at 2.399(1) and 2.419(1) Å at one side, and at 2.405(1), and 2.416(1) Å at the other side of the complex which are significantly longer than those observed earlier (between 2.276(2) and 2.348(3) Å).[494-507]

In the structure **6.4**, the shortest Fe-N bond lengths are the Fe-N bonds to the central triazine rings in tpt ligands. This observation is consistent with the terpyridyl-like metal complexes.[186,260,332,348,508-510]

There are also two water molecules in the asymmetric unit. One of the water molecules is highly disordered. Satisfactory results were obtained when one full-occupancy O atom, one 0.7-occupancy O atom, and one 0.3-occupancy O atom were defined in the asymmetric unit. However, the anisotropic displacement parameters of the disordered O atoms were large. Several trials at defining additional sites or using lower site-occupation factors for the disordered O atoms yielded inferior results. Indeed, omitting the disordered water molecule would result in an incomplete refinement with a higher R factor (≈ 6% *vs* 4%).

The hydrogen atoms on the disordered water molecule could not be located in the difference Fourier maps. The ordered water hydrogen atoms were, however, found in difference electron density maps and their positions were refined with a restrained geometry [O-H = 0.85(1) Å; with U_{iso}(H) = 1.2U_{eq}(O)].

There is an extensive hydrogen bonding network involving the ordered water molecules and the coordinated chloride ions. There are also π-π stacking interactions between the neutral molecules in the structure (Figure 6.4).

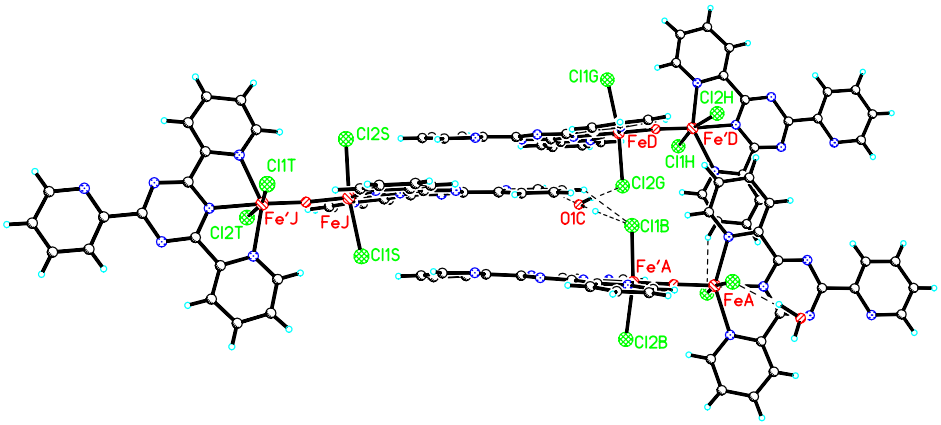


Figure 6.4. Hydrogen bonding and π-π stacking interactions within the crystal lattice of the complex **6.4**.

There are two kinds of π-π stacking interactions (face-face) between the adjacent neutral molecules: (1) π-π stacking interactions between the flanking pyridyl rings of the adjacent neutral molecules with the separation of 3.89 Å (face-face, centroid–centroid) (Figure 6.5); (2) π-π stacking interactions between the central triazine ring of one molecule and the end pyridyl ring of the adjacent molecule. The separation is 4.01 Å (face-face, centroid-centroid) (Figure 6.6).

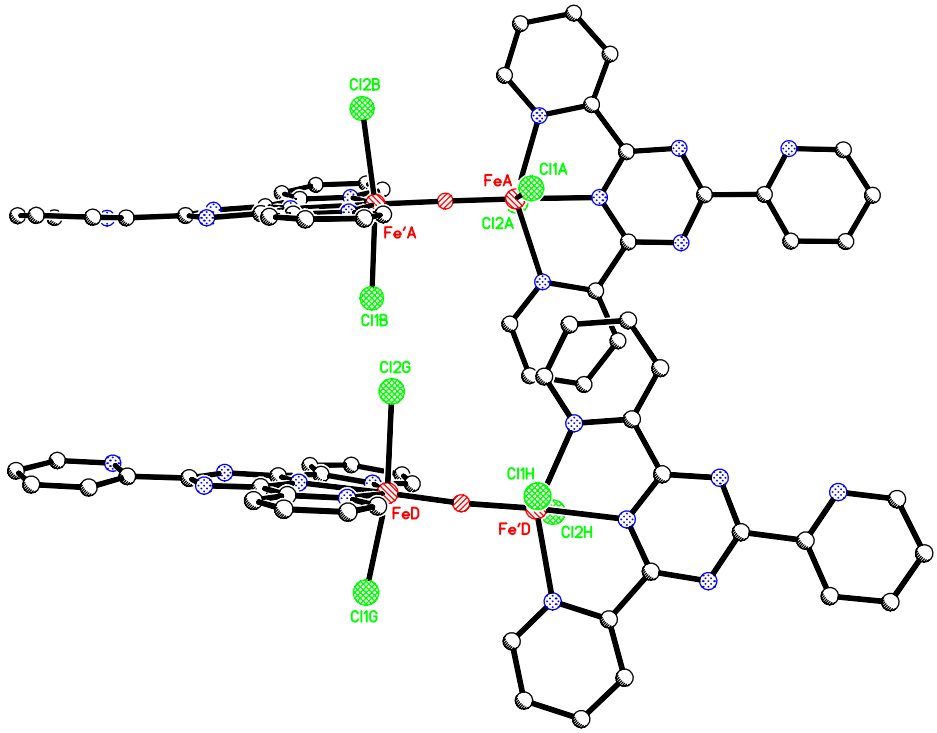


Figure 6.5. π-π stacking interactions between the flanking pyridyl rings of the adjacent neutral molecules. The separation distance between the flanking pyridyl rings is 3.89 Å (centroid–centroid).

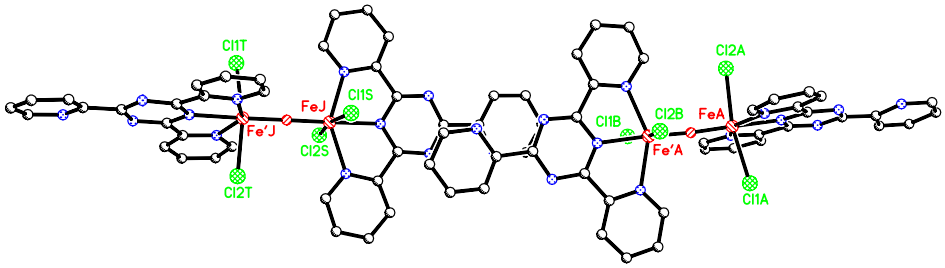


Figure 6.6. π-π stacking interactions between the central triazine ring of one molecule and the end pyridyl ring of the adjacent molecule. The separation is 4.01 Å (face-face, centroid-centroid).

6.4. Structure of Hexaaquacopper(II) Dinitrate Complex

inorganic papers

Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Hexaaquacopper(II) dinitrate: absence of Jahn–Teller distortion

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Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(O–N) = 0.004 Å
R factor = 0.034
wR factor = 0.096
Data-to-parameter ratio = 9.0

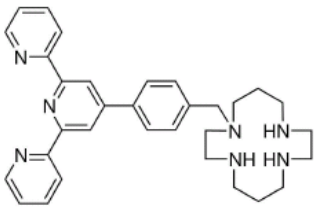
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 25 November 2005
Accepted 14 December 2005
Online 21 December 2005

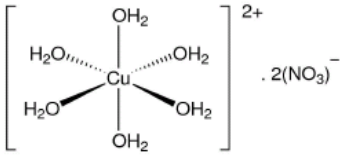
In the title compound, $[Cu(H_2O)_6](NO_3)_2$, the geometry around the Cu^{II} ion is approximately octahedral, formed by six O atoms from the coordinated water molecules. The Cu—O distances are rather similar [2.014 (2)–2.084 (2) Å] and not related by symmetry. The Jahn–Teller effect is, at best, only weakly observed in this structure, in contrast to many other structures where the hexaaquacopper(II) ion has been characterized. An extensive mesh of hydrogen-bond interactions between the coordinated water molecules and nitrate ions is a feature of the structure and may limit the degree to which the Jahn–Teller effect can be observed.

Comment

During attempts to grow crystals of a copper complex of the ditopic ligand, 1-[4′-*p*-tolyl-(2,2′:6′,2″-terpyridyl)]-1,4,8,11-tetraazacyclotetradecane, (I) (Padilla-Tosta *et al.*, 2000), blue block-shaped crystals of $[Cu(H_2O)_6](NO_3)_2$, (II), formed instead from the reaction mixture. Attempts to grow similar crystals in the absence of the ditopic ligand proved unsuccessful, which leads us to speculate that the ditopic ligand may be influencing the crystallization process. Unfortunately, the vagaries of nucleation and crystal growth make it difficult to test this hypothesis. We report here the structure of the hexaaquacopper(II) complex as its dinitrate salt.



(I)



(II)

The asymmetric unit of (II) consists of a $[Cu(H_2O)_6]^{2+}$ cation and two nitrate anions. The geometry around the Cu^{2+} can be best described as octahedral, with bonds to six water molecules (Fig. 1 and Table 1). The Cu—O bond lengths are rather similar, falling in the range 2.014 (2)–2.084 (2) Å, and there is an extended hydrogen-bonding network that links the coordinated water molecules and the nitrate anions throughout the crystal structure (Fig. 2 and Table 2). Bond lengths and angles in the nitrate anions [1.233 (4)–1.272 (4) Å and 118.5 (3)–121.1 (3)°, respectively] are unremarkable, there being only small deviations from the ideal geometry.

The similarity of the Cu—O bond lengths is rather unusual in that Jahn–Teller distortion often leads to two of the copper-ligand bonds that lie along one axis being much longer than

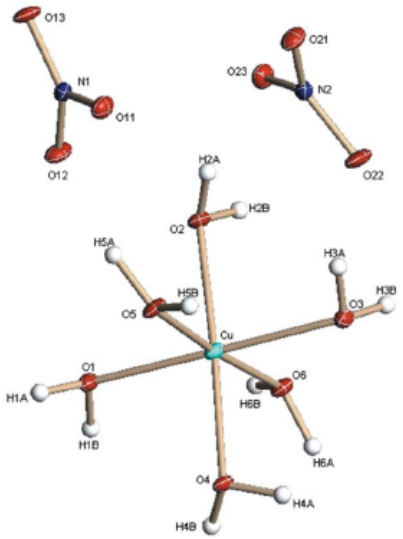


Figure 1
A perspective view of the title Cu^{II} complexes, (II), showing the atom-labelling scheme with 50% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

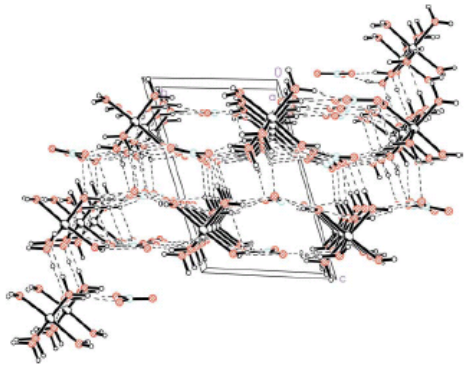


Figure 2
A packing diagram, showing hydrogen-bonding interactions (dashed lines) within the crystal structure of the complex. Key: Cu, N, O, and H atoms are shown with light blue (large), purple, red, and light blue (small) circles, respectively.

the remaining four copper–ligand bonds. A number of Jahn–Teller-distorted hexaaquacopper(II) complexes have been characterized by X-ray crystallography, *viz.* X–3($C_2H_{10}N_2^{2+}$)–2($O_{12}P_4^{4-}$) (Averbuch-Pouchot & Durif, 1989), X–2(ClO_4^-)–2($C_6H_{10}N_2O_2$) (Benedetti *et al.*, 1986), X–2($C_6H_4ClO_3S^-$) (Bernardinelli *et al.*, 1991), X–2($C_7H_7O_3S^-$) (Couldwell *et al.*, 1978), X–2($C_9H_9O_9S_3^{3-}$)–1.3(H_2O) (Dalrymple *et al.*, 2002), X–2($C_2H_{10}N_2^{2+}$)–$O_{18}P_6^{6-}$ (Durif & Averbuch-Pouchot, 1989), X–$C_6H_8CuO_{10}^{2-}$ (Filippova, 2000), X–2($C_{12}H_{10}O_4P^-$)–2($C_2H_5NO_2$) (Glowiak & Podgorska, 1986), X–$C_{16}H_{16}CuO_{10}^{2-}$ (Honghui *et al.*, 1988), X–$C_{16}H_{16}CuO_{10}^{2-}$ (Kennard & Smith, 1989), X–2(Cl_4^-),2(H_2O) (Li *et al.*, 2004), X–2 Cl^-–2($C_{10}H_8N_2O_2$)–2(H_2O) (Ma *et al.*, 2001), X–2($C_7H_5O_6S^-$)–2(H_2O) (Ma *et al.*, 2003), X–2(NH_4^+)–2(SO_4^{2-}) (Maslen *et al.*, 1988), X–2($C_{24}H_{44}H_{16}O_4Pt_4^{4+}$)–10($ClO_4^-$)–9($H_2O$) (Navarro *et al.*, 2000), X–($C_6H_8CuO_{10}^{2+}$) (Rodriguez-Martin *et al.*, 2002), X–2($C_8H_{11}N_4O^+$)–2(SO_4^{2-})–2(H_2O) (Shamuratov *et al.*, 1993) and $(X)_n$–2n($C_5H_8O_4^-$)–4n(H_2O) (Zviedre *et al.*, 1985), where X is $[Cu(H_2O)_6]^{2+}$. In these cases, the axial Cu—O bond lengths fall in the range 2.202–2.423 Å, in comparison with the equatorial bond lengths (1.945–2.084 Å). The mean axial bond length is between 8.7 and 24% longer than the mean equatorial bond length in these structures (the mean value of these percentage differences is 18.6% over 20 structures). In our structure, the mean bond length along the longest axis (O2—Cu—O4) is only 1.6% longer than that along the remaining axes.

We are aware of only six crystallographic studies of copper(II) complexes where static Jahn–Teller distortions are not observed in complexes where all six donors are otherwise identical, *viz.* in X–(BrO_3)$_2$ (Blackburn *et al.*, 1991), $Cu(en)_3^{2+}$–SO_4^{2-} (Cullen & Lingafelter, 1970), $2K^+$–Pb^{2+}–$Cu(NO_2)_6^{4-}$ (Cullen & Lingafelter, 1971), $Cu[[(CH_3)_2N]_2P(O)OP(O)[N(CH_3)_2]_2]_3(Cl_4)_2$ (Joesten *et al.*, 1970), X–$(SiF_6)^{2-}$–6(H_2O) (Ray *et al.*, 1973) and $2Tl^+$–Pb^{2+}–$Cu(NO_2)_6^{2-}$ (Takagi *et al.*, 1976), where X is $[Cu(H_2O)_6]^{2+}$. The structure we report further stands out from these other six because, in this case, the Cu atom lies on a general position, with all Cu—O bond lengths being independently refined. In the other six cases, the Cu atoms are located on special positions in higher symmetry space groups ($Pa\bar{3}$, $P\bar{3}1c$, $Fm\bar{3}$, $P\bar{3}c1$, $R\bar{3}$ and $Fm\bar{3}$, respectively).

Jahn–Teller distortion may not be observed in a crystallographic study if either there is disorder in the structure (so that a defined long axis is randomly distributed over the three orientations relative to the unit cell axes), or there is sufficient thermal motion to allow the long and short bonds in a structure to exchange over time (sometimes referred to as the dynamic Jahn–Teller effect). In these cases, the averaging inherent in the X-ray experiment (over spatial location in the crystal in the first case or time in the second) might be expected to manifest itself in the crystallographic modelling process as larger than expected anisotropic displacement parameters for the donor atoms along the direction of the copper–ligand bond. This effect has been discussed (Cullen *et al.*, 1970) and may be significant in a number of the literature cases (Blackburn *et al.*, 1991; Cullen *et al.*, 1971; Takagi *et al.*, 1976). Table 3 presents the anisotropic displacement parameters of Cu and the water O atoms in the structure of (II). The largest principal axes of the ellipsoids are not directed along the Cu—O bonds (Fig. 1). Taken together, these data strongly suggest the lack of Jahn–Teller distortion (static or dynamic) in the structure of (II). Here, three marginally longer Cu—O bonds (Cu—O2, Cu—O3 and Cu—O4) are meridionally distributed around the Cu atom, as are the Cu—O shorter bonds. The variation in the Cu—O bond lengths of the structure, and the absence of any significant Jahn–Teller effect, may be explained by the influence of the hydrogen-bonding network in the crystal structure of the complex (Fig. 3

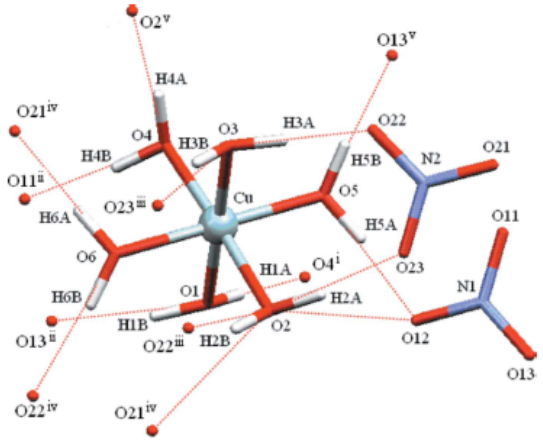


Figure 3
A perspective view of the Cu^{II} complex, (II), showing the hydrogen-bonding interactions (dashed lines) involving the dication. [Symmetry codes: (i) $-x, -y, -z$; (ii) $x-1, y-1, z$; (iii) $-x+1, -y+1, -z+1$; (iv) $x, y-1, z$; (v) $x-1, y, z$.]

and Table 2). All of the coordinated water molecules are involved in several hydrogen bonds, which means that, while the copper centre may not be in its lowest energy Jahn–Teller distorted state, this could be made up for by the large number of weak interactions that may each be marginally stronger in the less distorted structure.

Experimental

A solution of $Cu(NO_3)_2{\cdot}3H_2O$ (50 mg) in ethanol (5 ml) was added to a cooled filtered solution of ligand *L*, (I) (0.15 g), in ethanol (5 ml). The reaction mixture was heated at reflux for 1 h, and, upon cooling to room temperature, afforded a blue–green insoluble precipitate (0.11 g). The precipitate was suspended in ethanol–water (1:1, 5 ml), then the mixture was filtered after it was heated to reflux for 1 h. The solution was allowed to cool to room temperature overnight. The solution was kept in a refrigerator for about two months during which time blue crystals of (II) suitable for X-ray analysis were produced. No crystals of (I) or its copper complex were produced in this way.

Crystal data

$[Cu(H_2O)_6](NO_3)_2$
$M_r = 295.67$
Triclinic, $P\overline{1}$
$a = 5.7404\ (8)$ Å
$b = 7.6452\ (10)$ Å
$c = 11.4655\ (15)$ Å
$\alpha = 106.428\ (2)°$
$\beta = 98.399\ (2)°$
$\gamma = 101.504\ (2)°$
$V = 461.84\ (11)$ Å^3
$Z = 2$
$D_x = 2.126$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2722 reflections
$\theta = 2.9–26.4°$
$\mu = 2.43$ mm^{-1}
$T = 93\ (2)$ K
Block, blue
0.55 × 0.34 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.341$, $T_{max} = 0.744$
2917 measured reflections
1556 independent reflections
1494 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.1°$
$h = -6 \rightarrow 6$
$k = -8 \rightarrow 8$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.096$
$S = 0.91$
1556 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 2.7318P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -1.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu—O5	2.014 (2)	N1—O11	1.241 (4)
Cu—O1	2.034 (2)	N1—O12	1.245 (4)
Cu—O6	2.041 (2)	N1—O13	1.268 (4)
Cu—O4	2.064 (2)	N2—O21	1.233 (4)
Cu—O3	2.074 (2)	N2—O23	1.252 (4)
Cu—O2	2.084 (2)	N2—O22	1.272 (4)
O5—Cu—O1	89.49 (10)	O6—Cu—O3	85.17 (10)
O5—Cu—O6	175.94 (10)	O4—Cu—O3	92.35 (10)
O1—Cu—O6	93.58 (10)	O5—Cu—O2	89.50 (10)
O5—Cu—O4	91.38 (10)	O1—Cu—O2	87.93 (10)
O1—Cu—O4	88.81 (10)	O6—Cu—O2	87.96 (10)
O6—Cu—O4	91.34 (10)	O4—Cu—O2	176.61 (9)
O5—Cu—O3	91.72 (10)	O3—Cu—O2	90.89 (10)
O1—Cu—O3	178.31 (10)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O4^{i}	0.98 (2)	1.91 (2)	2.894 (3)	179 (3)
O1—H1*B*···O13^{ii}	0.97 (2)	1.79 (2)	2.741 (4)	168 (4)
O2—H2*B*···O22^{iii}	0.98 (2)	2.12 (2)	3.038 (4)	156 (3)
O2—H2*A*···O23	0.98 (2)	2.00 (2)	2.940 (4)	162 (3)
O2—H2*B*···O21^{iv}	0.98 (2)	2.38 (3)	2.912 (4)	113 (3)
O3—H3*A*···O22	0.98 (2)	1.83 (2)	2.779 (4)	162 (3)
O3—H3*B*···O23^{iii}	0.97 (2)	1.88 (2)	2.827 (4)	167 (3)
O4—H4*A*···O2^{v}	0.97 (2)	1.99 (2)	2.942 (4)	167 (4)
O4—H4*A*···O1^{v}	0.97 (2)	2.60 (4)	3.070 (3)	110 (3)
O4—H4*B*···O11^{ii}	0.97 (2)	1.79 (2)	2.763 (4)	175 (3)
O5—H5*A*···O12	0.97 (2)	1.78 (2)	2.735 (3)	166 (4)
O5—H5*A*···N1	0.97 (2)	2.50 (3)	3.417 (4)	156 (3)
O5—H5*A*···O11	0.97 (2)	2.58 (3)	3.285 (3)	130 (3)
O5—H5*B*···O13^{v}	0.96 (2)	1.78 (2)	2.740 (4)	172 (4)
O5—H5*B*···N1^{v}	0.96 (2)	2.47 (2)	3.365 (4)	155 (3)
O5—H5*B*···O12^{v}	0.96 (2)	2.45 (3)	3.123 (3)	126 (3)
O6—H6*B*···O21^{iv}	0.98 (2)	2.44 (3)	3.154 (3)	130 (3)
O6—H6*B*···O22^{iv}	0.98 (2)	1.91 (2)	2.860 (4)	162 (4)
O6—H6*B*···N2^{iv}	0.98 (2)	2.51 (2)	3.436 (4)	157 (3)

Symmetry codes: (i) $-x, -y, -z$; (ii) $x-1, y-1, z$; (iii) $-x+1, -y+1, -z+1$; (iv) $x, y-1, z$; (v) $x-1, y, z$.

Table 3
Selected anisotropic displacement parameters (Å^2).

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu	0.0058 (3)	0.0106 (3)	0.0126 (3)	0.00344 (18)	0.00374 (17)	0.00079 (17)
O1	0.0078 (12)	0.0084 (13)	0.0119 (12)	0.0020 (10)	0.0045 (10)	0.0012 (9)
O2	0.0050 (11)	0.0102 (13)	0.0130 (12)	0.0047 (10)	0.0024 (9)	0.0003 (9)
O3	0.0091 (12)	0.0099 (13)	0.0123 (12)	0.0032 (10)	0.0053 (10)	0.0027 (10)
O4	0.0052 (11)	0.0079 (13)	0.0134 (12)	0.0031 (10)	0.0035 (9)	0.0013 (9)
O5	0.0067 (12)	0.0120 (13)	0.0160 (13)	0.0076 (10)	0.0064 (10)	0.0021 (10)
O6	0.0058 (12)	0.0114 (13)	0.0137 (12)	0.0063 (10)	0.0046 (10)	0.0014 (9)

H atoms were located in a difference Fourier map. The O—H distances were restrained to 1.00 (2) Å, with U_{iso}(H) = 1.2U_{eq}(O). In the final difference map the deepest hole is located 0.89 Å from the Cu atom.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *MERCURY* (Version 1.4; Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXTL*.

The authors thank Professor W. T. Robinson and Dr J. Wikaira for their help with X-ray structural investigations associated with this project. A University of Canterbury Doctoral Scholarship (to RZ) is gratefully acknowledged.

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6.5. Conclusion

X-ray crystal structures of three metal complexes were explained individually: (1) $[Ru(bppt)_2](PF_6)_2$, (2) $[Fe(ttp)(Cl)_2]_2(\mu\text{-}O).2H_2O$, and (3) $[Cu(H_2O)_6](NO_3)_2$.

In the first complex, the coordination of Ru is octahedral. The coordination sphere is formed by six N atoms from the tridentate sites of two ligands that are meridionally oriented around the Ru(II) ion. The locations of the donor atoms deviate substantially from an ideal octahedron. The Ru-N distances are in the range 1.983 (4)–2.111 (4) Å. The Ru atom occupies a special position with twofold rotational symmetry. One of the P atoms occupies a special position with twofold rotational symmetry and the other P atom is located on a special position at a centre of symmetry.

In the second complex, each Fe ion is coordinated to tridentate terpyridyl site of the ligand tpt in the complex. The fourth and the fifth coordination sites of each Fe ion is also occupied by two Cl ions *cis* to the oxo bridge. The last coordination sites of the Fe ions were linked together through an oxo bridge. The tpt ligands act as planar tridentate binding moiety while the remaining bidentate binding sites in the ligands remain non-coordinated. Bond angles suggest that octahedron at each Fe centre is distorted. The Fe-O-Fe angle is 176.12(15) Å. The Fe-Cl bond distances in this structure are at 2.3999(9) and 2.4187(9) Å at one side, and at 2.4051(10), and 2.4162(10) Å at the other side of the complex. There are some π-π stacking interactions as well as some hydrogen bonding ineractions by which the structure is stabilized.

In the last structure, the geometry around the Cu(II) ion is approximately octahedral, formed by six O atoms from the coordinated water molecules. The Cu-O distances are rather similar [2.014 (2)–2.084 (2) Å] and not related by symmetry. The Jahn–Teller effect is, at best, only weakly observed in this structure, in contrast to many other structures where the hexaaquacopper(II) ion has been characterised. An extensive mesh of hydrogen-bond interactions between the coordinated water molecules and nitrate ions is a feature of the structure and may limit the degree to which the Jahn–Teller effect can be observed.

Chapter 7

Conclusions and Future Prospects

This thesis has described the preparations of a number of new ditopic ligands with two different binding sites, one of which is a tridentate terpyridyl chelating unit. Some central triazine containing bridging ligands were also used in our studies. The coordination chemistry of these ligands has been investigated with a range of transition metal atoms, mainly with a focus on Ru(II) and Co(III) ions. Selective complexations were achieved by means of defining the geometries around the complex precursors.

In these studies, we mainly focussed on the preparation and use of these ditopic ligands where two metal ion binding sites are differentiated by the number of atoms in each site (such as bppt, **2.1**, and tpt, **1.70** in Chapter 2), the configuration of the binding site (such as pzt, **1.71**, in Chapter 4 and tcnt, **1.76**, in Chapter 5), or the types of donor atom that are present in the sites (such as bpat, **5.31**, dint, **5.33**, cymt, **1.74**, cynt, **1.75**, and tcnt, **1.76**, in Chapter 5) (Figure 7.1). This binding site differentiation allows us to use the different coordination properties of the binding sites to control the regiochemistry of the complexation, ensuring that the correct metal ion is incorporated at the correct binding site in the ligand.

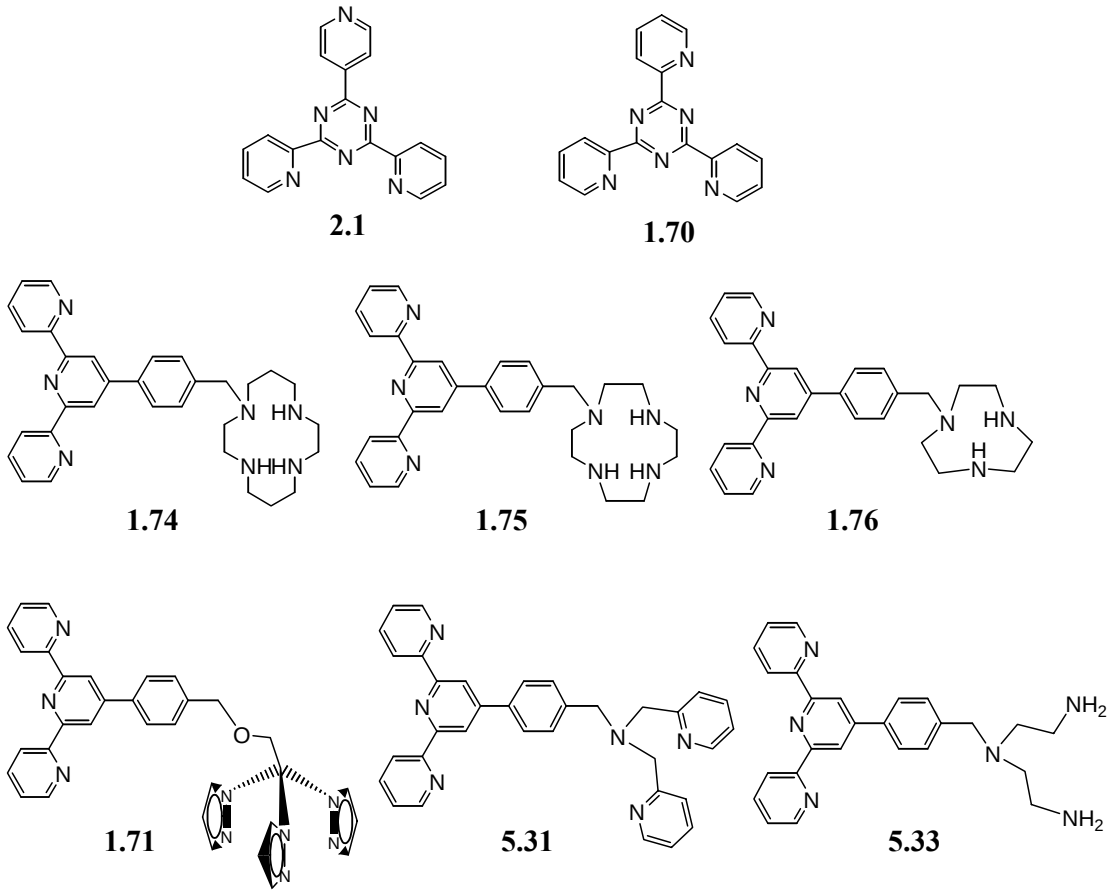


Figure 7.1. Some ditopic ligands based on terpyridyl systems.

Central triazine containing ligands bppt, **2.1**, and tpt, **1.70**, initially appealed to us mainly because of their improved photoluminescence properties in comparison with their terpy analogues. These ligands also provide two different binding sites, including tridentate sites suitable for binding to Ru(II) ions. Unfortunately, attempts to prepare dinuclear Ru(II)-Co(III) complexes of ligand **1.70** proved unsuccessful, presumably because of the steric hindrance around the bidentate binding domain in its Ru(II) complex.

Chapter 2, however, describes the preparation and crystallographic identification of a series of Ni(II) complexes of the triazine tpt ligand, **1.70**. We demonstrated that the tpt ligand can form a range of complexes with Ni(II) ions, depending on the other ligands, anions and solvents that are present in the reaction

mixture. While the formation of particular complexes or mixtures of complexes can be understood based on general principles of reactivity in labile systems, it is not possible to predict just which complex(es) will be isolated under a given set of conditions. We were able to synthesise and crystallographically characterise some dinuclear Ni(II) complexes of the tpt ligand. Reactions of the tpt ligand with Ru(II) and Fe(II) ions were also studied. In chapter 6, a crystal structure of an oxo-bridged iron complex of ligand tpt is described. The π-π stacking interactions within the lattice of the complex are a feature of the structure. Some ruthenium complexes of the related bppt ligand were also prepared and the crystal structure of a bis(bppt) complex was described in Chapter 6.

Ligand pzt, **1.71**, in Chapter 4, was initially one of the best candidates in our studies, as it contains a terpyridyl fragment and a tripodal tris(pyrazolyl) fragment, which may allow synthetic differentiation between the tridentate binding sites as a result of the expected *mer* and *fac* coordination preferences of these groups. Unfortunately, we found no evidence of any coordination to the tris(pyrazolyl) site when the ligand pzt, **1.71**, or its Ru(II) or Fe(II) complexes, **4.4**, **4.5**, and **4.6**, respectively, were treated with $CoCl_2$, $Na_3[Co(NO_2)_6]$, or $Co(NO_3)_2$. Therefore, our strategy for preparation a dinuclear complex containing Ru(II) and Co(III) using this ligand was unsuccessful!

However, in Chapter 4, the synthesis, characterisation, and coordination chemistry of pzt ligand are described. The coordination chemistry of this ligand revealed number of different architectures including dinuclear complexes, [2+2] metallomacrocycles and one-dimensional coordination polymers. NMR, X-ray crystallography, and ESI-MS were powerful tools for the study of this ligand and its coordination chemistry. Chemical forces including π-π stacking interactions by which

the lattices of the structures were stabilised, were significant in the crystal lattices of Ag(I), Cu(II), and Zn(II) complexes of pzt ligand; while the lack of π-π stacking interactions in the lattice of the ligand itself was also an interesting issue.

In order to conduct a model study to guide our work with a more complicated and less readily available pzt ligand, tripodal ligand tris(1*H*-pyrazol-1-yl)methane (tpm) was synthesised and its ruthenium complexes were studied in Chapter 3. Unfortunately, attempts for preparation of Co(III) complexes of tpm ligand were unsuccessful under our conditions. We also demonstrated that $[Ru(tpm)_2]^{2+}$ complexes could not be formed under our conditions, as one of the pyrazolyl rings of the second tpm ligand always remained uncoordinated. Other available coordinating monodentate ligands/solvent molecules in the solution occupied the sixth coordination site of the complex instead. In $[Ru(tpm)_2Cl]^{+}$, for example, one tpm ligand is found to coordinate in a *fac* tridentate manner, while the second tpm ligand acts as a bidentate ligand. The pendant pyrazolyl group of this second tpm ligand could not be induced to coordinate, even when the chloride ligand was removed by reaction with silver(I) ions.

While the coordination chemistry of Ru(II) complexes of tris(pyrazolyl)methane ligands has been studied, it appeared that attachment of such a group to another metal ion binding site significantly reduces its ability to bind metal ions. Presumably the positive charge on the ligand that results from coordination of a metal ion in the terpyridyl site reduces the ability of the tris(pyrazolyl)methane site to coordinate to other metal ions. We have found, however, that silver ions coordinate to both sites in the pzt ligand and a dinuclear $[Ag_2(pzt)_2]^{2+}$ complex, **4.2**, is formed when the silver ions and ligands are present in high concentration. Silver ions could also be used to link together pzt complexes of other metal ions to form polymeric species.

Presumably this is a result of both the low charge on the silver ion and the ability of silver ions to bind to heterocyclic nitrogen donors.

Ditopic ligands bpat, **531**, dint, **5.33**, tcnt, **1.76**, pcymt, **5.35**, cymt, **1.74**, and cynt, **1.75**, (in Chapter 5) were prepared and their Ru(II) complexes were synthesised. Two main approaches were employed for the preparation of the heterodinuclear complexes using these ligands: (1) "direct coordination"; in which the ditopic ligand was allowed to react to the Ru(III) complex precursor, **3.2**, under mild reduction conditions, followed by complexation of Co(III) ions in the second site. (2) "protection-complexation-deprotection", in which a protected ligand was used. When a ligand contains a binding site which is very similar to its terpyridyl site, the protected analogue of the ligand was used to react with Ru(II) complex **3.2**. Further complexation to Co(III) ion at the other site of the ligand was achieved after deprotection of the (non coordinated) ligand.

Synthetic strategies for preparations of the ditopic ligands and their heterodinuclear complexes were discussed in Chapter 5. Ru(II)-Co(III) dinuclear complexes of a selected ligand cymt, **1.74**, were prepared. They were characterised using techniques such as NMR, ESI-MS, and IR spectroscopy. The bidentate ligand en was used as a close structural analogue for nitrogen mustards (see Chapter 1) to react with the Ru(II)-Co(III) complex **5.46**. Unfortunately, attempts to synthesise the target molecule **5.47** (in Chapter 5) failed under a variety of conditions.

When an excess en ligand in MeOH was used, the parent complex **5.30** in which the cobalt ion has been removed from the macrocycle was regenerated in 98% yield. The reasons for this are not understood at this stage; but it may be related to the high charge on the complex. Under milder conditions, some evidence of coordination of the ligand was observed. The en ligand may be acting as a monodentate ligand *trans* to the

coordinated Cl^- ion in the complexes. The other end of the en ligand may be protonated. Protonation of the en ligand would presumably inhibit its ability to act as a bidentate ligand in the complexes.

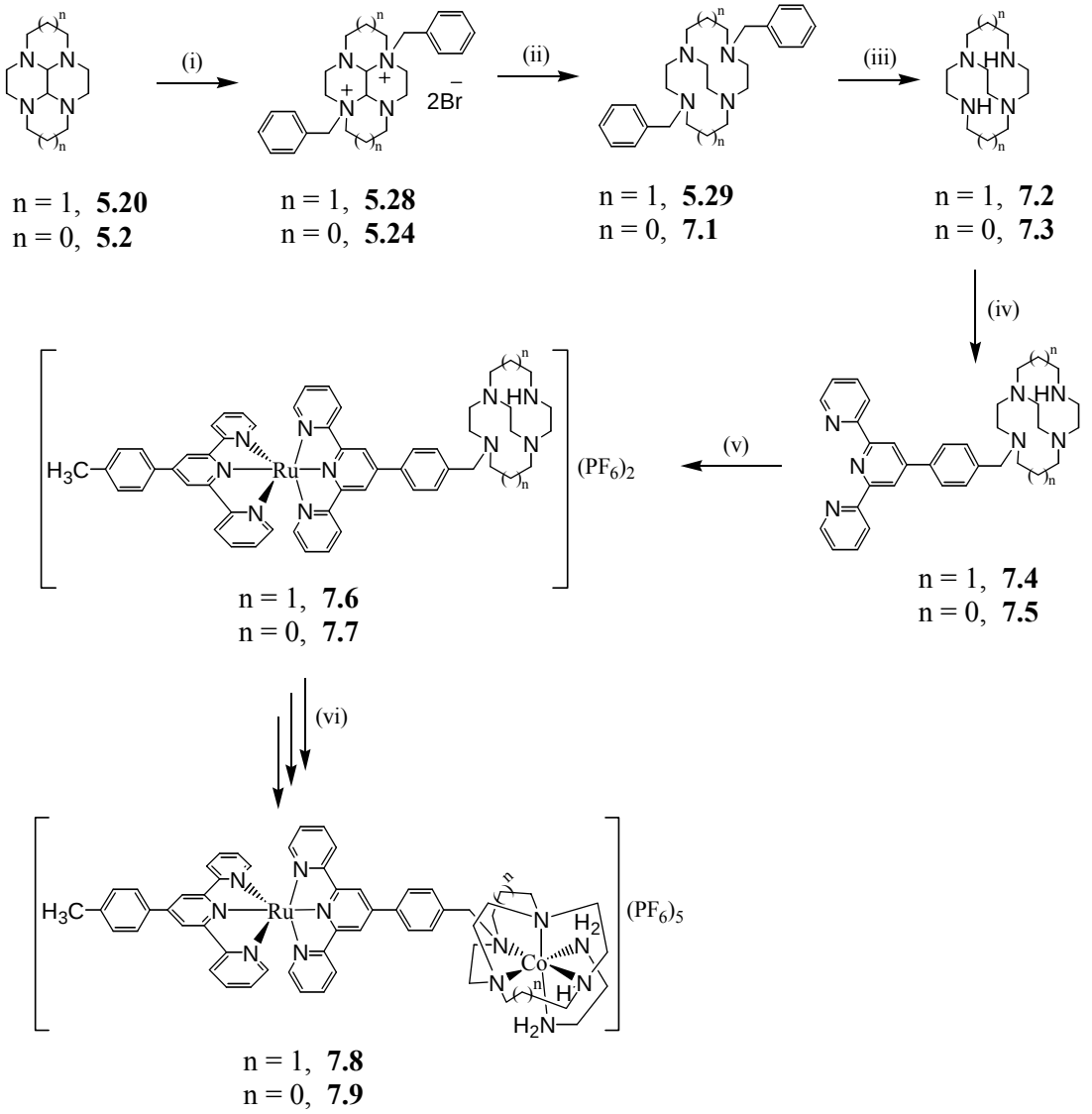
In summary, the Ru(II) complexes containing bridging ligands, can be classified into three main categories depending on the type of ligands that have been employed: (1) Ru(II) complexes which can react with Co(III) ions to form heterodinuclear Ru(II)-Co(III) complexes; (2) Ru(II) complexes which react only with Ag(I) ions and no other common metal ions that we have tried; (3) Ru(II) complexes with no detectable ability to coordinate other common metal ions.

Our study in Chapter 5 demonstrated a synthetic methodology by which Ru(II)-Co(III) complexes of the type $[(ttp)Ru(cymt)Co(X)_2]^{3+}$; where X = NO_2^-, Cl^-, and OH^-, can be efficiently prepared from the corresponding Ru(II) complexes. However, getting a bidentate ligand that can be detected when it is released, attached to Co(III) ions of these complexes is still a synthetic challenge. Reactions of heterodinuclear Ru(II)-Co(III) complexes with en ligands result in removal of the cobalt ions from the complexes. This is may be a result of a significant difference in the overall charges between the complexes with anionic and the complexes with neutral ligands (3+ *vs* 5+) that may lead to destabilization of the complexes. Higher overall charges of complexes when protonable ligands such as monodentate en are present, may destabilize the complexes even more.

Using more rigid and defined acceptor binding sites in the bridging ligands may be useful in the future. More rigid macrocycles may increase the stability of the complexes and at the same time reduce the number of stereoisomers. Cross-bridged cyclams/cyclens are relatively more rigid binding sites than their parent systems, cyclam/cyclen. These systems also form stable and robust complexes and can be

readily attached to the other molecules (such as bromo compound btp) by nucleophilic substitution reaction. These cross-bridged systems are also potentially suitable candidates to react with Co(III) ions in *cis*-folded geometries. This approach may facilitate Co(III) ions to bound with bidentate ligands in desired *cis* arrangements.

A multi-step method for preparation of dinuclear Ru(II)-Co(III) complex analogues of **4.59** (in Chapter 5) is proposed in Scheme 7.2. By following this method, in future, one could, hopefully, be able to synthesise a modified heterodinuclear complex with reduced possible stereoisomers.



Scheme 7.2. Proposed methods for preparation of heterodinuclear Ru(II)-Co(III) complexes: (i) excess benzylbromide, CH_3CN, r.t, 14 days;[295] (ii) 1. excess $NaBH_4$,

95% EtOH, r.t, 16 days; 3 M HCl, then remove EtOH; 2. KOH, H_2O, benzene;[295] (iii) 1. H_2 (1 atm.), 10% Pd/C, HOAc, r.t; 2. HOAc removal, then KOH, H_2O, benzene;[293] (iv) **1.50**, CH_2Cl_2, 30ºC, 12-24 h; (v) **3.2**, MeOH, *N*-methylmorpholine, reflux, 1-2 h; NH_4PF_6; (vi) 1. $Na[Co(NO_2)_6]$, acetone:water (2:1), 50ºC, 4-6 h in dark; 2. 6 M HCl, steam bath in dark; 3. en, 30ºC, 6h in dark.

Once heterodinuclear Ru(II)-Co(III) complexes of this kind have been made, in future, then their ligand release upon irradiation by light of a particular wavelength will be studied. If it can be demonstrated that photoinduced release of DNA alkylating agents (such as nitrogen mustards, Chapter 1) takes place, then those complexes can be considered for biological evaluation. Full characterisation of such reactions will require detailed photophysical studies and study of the relevant redox chemistry, both for molecules containing cytotoxins and model compounds such as those prepared during this project.

Chapter 8

Experimental

8.1. General Experimental

Reagent-grade solvents were used in syntheses of the Ni complexes without further purification. All solvents used in syntheses of other complexes were dried and distilled according to the standard methods[511] prior to use. All dried and distilled solvents used in reactions of air sensitive compounds (in Chapters 3-6) were deareated prior to use by saturation with argon. Syntheses of ditopic ligands and their complexes were carried out under an inert atmosphere of argon using standard Schlenk techniques.

^{1}H NMR and ^{13}C NMR spectra were recorded on Varian UNITY-300 or Varian INOVA-500 spectrometers. ^{1}H NMR and ^{13}C NMR chemical shifts are referenced to residual solvent resonances or using TMS as an internal reference. ^{1}H NMR spectra were assigned using 2D COSY and NOESY techniques. Infrared spectra (400-4000 cm^{-1}) were obtained using a Shimadzu 8201PC Series FTIR interfaced with an Intel 486 PC operating Shimadzu HyperIR software. Spectra were obtained using diffuse reflectance method in solid KBr. Microanalyses were performed at the University of Otago. Solutions (10 μg/mL) for electrospray ionization mass spectrometry (ESI-MS)

were recorded using milliQ water or reagent grade DMSO and MeOH in Micromass LCT Waters 2795 Mass Spectrometer. UV-vis spectra were recorded on a Varian CARY Probe 50 UV-vis Spectrophotometer.

The ligand 2,4,6-tris(2-pyridyl)-1,3,5-triazine (tpt), **1.70**, was prepared according to the literature methods.[1] The ligands tris(1*H*-pyrazol-1-yl)methane (tpm), **3.1**,[263] 2,2,2-tris(1*H*-pyrazol-1-yl)ethanol (tpe), **4.1**,[263] 4'-(*p*-tolyl)-2,2':6',2''-terpyridine (ttp), **1.48**,[217,218,226] and 4'-(*p*-bromomethylphenyl)-2,2':6',2''-terpyridine (btp), **1.50**,[217,218] 1,5-bis(phthalimido)-3-azapentane,[469] *N,N′*-bis(2-pyridylmethyl)amine,[464] 1,4,8,11-tetraazacyclotetradecane[459] (cyclam), *cis*-decahydro-1*H*,6*H*-3a,5a,8a,10a-tetraazapyrene[480] (capped-cyclam), 1,4,7,10-tetraazacyclododecane[462,463] (cyclen), 1,4,7-triazacyclononane[462,463] (tacn), 1,4,7-triazatricyclodecane[479] (capped-tacn), 4′-(*p*-(1,4,7-triazacyclonon-1-yl)methylphenyl)-2,2′:6′-2″-terpyridine,[220] were prepared by following literature methods. 1,2-Bis-(4-pyridyl)-ethylene (bpe) was used as received from Aldrich. Pyrazole was obtained from Aldrich and used without further purification. Benzoyl peroxide (BPO) and *N*-bromosuccinimide (NBS) were purchased from Aldrich and recrystallized using standard methods and dried over P_2O_5 and under vacuum prior to use.

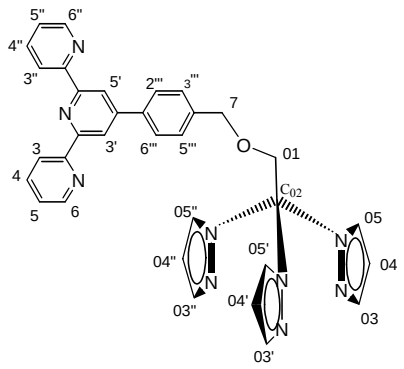
Complexes [Ru(tpm)Cl_3], **2**,[194] [Ru(tpm)(bpy)Cl]Cl, **9**,[194,398] [Ru(tpm)(bpy)(OH_2)](ClO_4)$_2$, **10**,[194] [Ru(tpm)(bpy)(bpe)](PF_6)$_2$, **11**,[397] and [Ru(ttp)Cl_3], **12**,[386] [Co(tacn)$(Cl)_3$], [Co(dien)$(Cl)_3$], [Co(trien)$(Cl)_2$], [Co(tren)$(Cl)_2$], [Co(NH_3)$_5$(OH_2)] were synthesized according to the literature methods. All other starting materials were obtained commercially and used without further purification.

All other starting materials were obtained commercially and used without further purification.

CAUTION: Perchlorate salts of metal complexes containing organic ligands are potentially explosive and should be handled with care and in small quantities.

8.2. Syntheses of Ligands

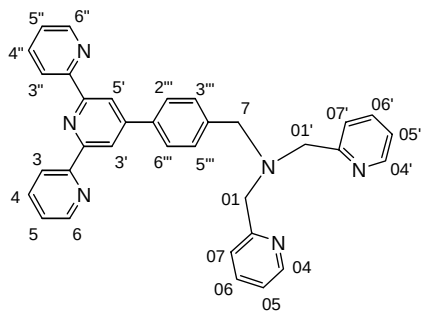
4'-(*p*-(2,2,2-tris(1*H*-pyrazol-1-yl)ethoxymethylphenyl)-2,2':6',2''-terpyridine (pzt), **1.71**



The solution of 2,2,2-tris(1*H*-pyrazol-1-yl)ethanol (tpe), **4.1**,[263] (0.368 g, 1.505 mmol) and 4'-(*p*-bromomethylphenyl)-2,2':6',2''-terpyridine (btp), **1.50**,[217,218] (0.605 g, 1.505 mmol) in dry THF (30 mL) was added dropwise to a suspension of NaH (0.15 g) in dry THF (20 mL) under argon over a period of 3 h. The mixture was stirred at reflux under argon for 24 h and then allowed to cool at room temperature. To this yellow solution was added enough water dropwise carefully to consume the excess NaH. The mixture was extracted from diethyl ether (4 × 50 mL), and the combined organic extracts were washed with 50 mL saturated $NaHCO_3$ solution, then with 50 mL saturated NaCl solution, and finally with 50 mL water. To the aqueous solution was added diethyl ether. The organic layer was extracted. After drying the combined extracts with anhydrous $MgSO_4$, filtered and the solvent was removed under reduced pressure to yield a pale yellow powder; mp 183-184˚. Purification was achieved by recrystallisation of the crude material from CH_2Cl_2-hexanes (1:5) to afford (0.7190 g, 84%) as a pale yellow microcrystalline material. Crystals suitable for X-ray diffraction were obtained by layering a dichloromethane solution of the ligand with hexanes. ^1H

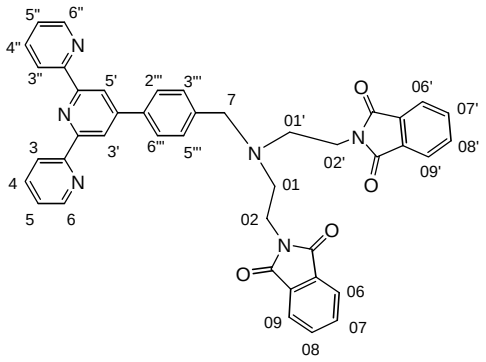
NMR (500 MHz; solvent $CDCl_3$): δ 8.734 (2H, d, H_6, $H_{6''}$), 8.727 (2H, s, $H_{3'}$, $H_{5'}$), 8.67 (2H, d, H_3, $H_{3''}$), 7.89 (2H, m, H_4, $H_{4''}$), 7.86 (2H, d, $H_{2'''}$, $H_{6'''}$), 7.68 (3H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.46 (3H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.36 (2H, m, H_5, $H_{5''}$), 7.30 (2H, d, $H_{3'''}$, $H_{5'''}$), 6.36 (3H, m, H_{04}, $H_{04'}$, $H_{04''}$), 5.20 (2H, s, H_{01}), 4.61 (2H, s, H_7). ^{13}C NMR (75 MHz; solvent $CDCl_3$): δ 156.12, 155.86, 149.81 (C_3, $C_{3''}$), 149.06, 141.35 (C_{03}, $C_{03'}$, $C_{03''}$), 138.07, 138.04 (C_4, $C_{4''}$), 136.94, 130.90 (C_{05}, $C_{05'}$, $C_{05''}$), 128.13 ($C_{3'''}$, $C_{5'''}$), 127.38 ($C_{2'''}$, $C_{6'''}$), 123.86 (C_5, $C_{5''}$), 121.38 (C_6, $C_{6''}$), 118.81 ($C_{3'}$, $C_{5'}$), 106.55 (C_{04}, $C_{04'}$, $C_{04''}$), 89.84 (C_{02}), 73.80 (C_7), 73.63 (C_{01}). ^{1}H NMR (500 MHz; solvent dmso-d_6, see Figure above for labeling): δ 8.86 (2H, d, H_3, $H_{3''}$), 8.80 (2H, s, $H_{3'}$, $H_{5'}$), 8.77 (2H, d, H_6, $H_{6''}$), 8.14 (2H, dd, H_4, $H_{4''}$), 7.99 (2H, d, $H_{2'''}$, $H_{6'''}$), 7.79 (3H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.63 (5H, m, H_{05}, $H_{05'}$, $H_{05''}$, H_5, $H_{5''}$), 7.48 (2H, d, $H_{3'''}$, $H_{5'''}$), 6.53 (3H, m, H_{04}, $H_{04'}$, $H_{04''}$), 5.17 (2H, s, H_{01}), 4.74 (2H, s, H_7).^{13}C NMR (75 MHz; solvent dmso-d_6) δ155.86, 155.07, 149.53 (C_3, $C_{3''}$), 149.33, 141.07 (C_{03}, $C_{03'}$, $C_{03''}$), 139.00, 137.71 (C_4, $C_{4''}$), 136.99, 131.23 (C_{05}, $C_{05'}$, $C_{05''}$), 128.56 ($C_{3'''}$, $C_{5'''}$), 127.07 ($C_{2'''}$, $C_{6'''}$), 124.77 (C_5, $C_{5''}$), 121.15 (C_6, $C_{6''}$), 118.07 ($C_{3'}$, $C_{5'}$), 106.65 (C_{04}, $C_{04'}$, $C_{04''}$), 89.42 (C_{02}), 72.80 (C_{01}), 72.46 (C_7). IR (KBr): $\tilde{\nu}$/cm^{-1} = 3140 m, 3055 m, 3017 m, 2939 m, 2885 w, 1983 w, 1929 w, 1790 w, 1728 w, 1697 w, 1582 m, 1520 m, 1466 m, 1389 s sh, 1312 s, 1258 m, 1196 m, 1126 s, 1096 s, 1042 w, 988 m, 949 w, 872 s sh, 895 m, 764 s, 656 m, 617 m, 525 m sh. ESI-MS: *m/z* 566.2931 ([M+H]$^+$, 100%). Anal. Calc. for $C_{33}H_{27}N_9O.0.5H_2O$ (574.65): C 68.98, H 4.91, N 21.94%; found: C 69.32, H 4.79, N 21.77%.

4'-(*p*-(*N*,*N'*-bis(2-pyridylmethyl)amine)-*N*"-methylphenyl)-2,2':6',2"-terpyridine (bpat**), 5.31**



A mixture of (*N*,*N'*-bis(2-pyridylmethyl)amine),[464] (0.24 g, 1.2 mmol) and 4'-(*p*-bromomethylphenyl)-2,2':6',2"-terpyridine (btp), **1.50**, (0.4 g, 1.0 mmol), and anhydrous K_2CO_3 (1.4 g, 10 mol) in dry CH_3CN (50 mL) was stirred at 55-60° C under argon for 3 days. The progress of the reaction was monitored by TLC. The reaction mixture was then allowed to cool at room temperature; filtered and the solvent was removed from the filtrate under reduced pressure to give a pale yellow oil. Purification by column chromatography (Al_2O_3, 1-2% MeOH in CH_2Cl_2) afforded the desired product (0.52 g, 96%) as a pale yellow oil. ^{1}H NMR (500 MHz; solvent $CDCl_3$): δ 8.729 (2H, s, $H_{3'}$, $H_{5'}$), 8.725 (2H, d, H_6, $H_{6"}$), 8.67 (2H, d, H_3, $H_{3"}$), 8.54 (2H, d, H_{04}, $H_{04'}$), 7.87 (2H, m, H_4, $H_{4"}$), 7.86 (2H, d, $H_{2'''}$, $H_{6'''}$), 7.69 (2H, m, H_{06}, $H_{06'}$), 7.61 (2H, d, H_{07}, $H_{07'}$), 7.56 (2H, d, $H_{3'''}$, $H_{5'''}$), 7.35 (2H, m, H_5, $H_{5"}$), 7.17 (2H, m, H_{05}, $H_{05'}$), 3.86 (4H, s, H_{01}, $H_{01'}$), 3.77 (2H, s, H_7). ^{13}C NMR (75 MHz; solvent $CDCl_3$): δ 159.61, 156.24, 155.87, 150.03, 149.09 (2C, C_6, $C_{6"}$), 148.98 (2C, C_{04}, $C_{04'}$), 140.18, 137.23, 136.83 (2C, C_4, $C_{4"}$ or $C_{2'''}$, $C_{6'''}$), 136.46 (2C, C_{06}, $C_{06'}$), 129.35 (2C, $C_{3'''}$, $C_{5'''}$), 127.26 (2C, C_4, $C_{4"}$ or $C_{2'''}$, $C_{6'''}$), 123.78 (2C, C_5, $C_{5"}$), 122.85 (2C, C_{07}, $C_{07'}$), 122.00 (2C, C_{05}, $C_{05'}$), 121.31 (2C, C_3, $C_{3"}$), 118.74 (2C, $C_{3'}$, $C_{5'}$), 60.06 (2C, C_{01}, $C_{01'}$), 58.20 (C_7). ESI-MS: *m/z* 261.1370 ($[M+2H]^{2+}$, 100%), 521.2880 ($[M+H]^+$, 14%).

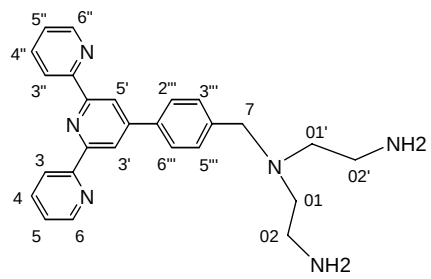
4'-(*p*-(1,5-bis(phthalimido)-3-azapentane)methylphenyl)-2,2':6',2''-terpyridine (bptt), **5.32**



A mixture of 1,5-bis(phthalimido)-3-azapentane,[469] (0.43 g, 1.2 mmol) and 4'-(*p*-bromomethylphenyl)-2,2':6',2''-terpyridine (btp), **1.50**, (0.4 g, 1.0 mmol), and anhydrous K_2CO_3 (1.4 g, 10 mmol) in dry CH_3CN (50 mL) was stirred at 55-60° C under argon for 3 days. The progress of the reaction was monitored by TLC. The reaction mixture was then allowed to cool at room temperature; filtered and the solvent was removed from the filtrate under reduced pressure to give a pale yellow oil. To the crude product was added water (100 mL); then was extracted with CH_3Cl (3 × 100 mL). After drying over $MgSO_4$, it was filtered and the solvent was removed from the filtrate to afford the analytically pure desired product (0.67 g, 98%) as a non-solid material. ^{1}H NMR (500 MHz; solvent $CDCl_3$): δ 8.77 (2H, d, H_6, $H_{6''}$), 8.68 (2H, d, H_3, $H_{3''}$) ,8.60 (2H, s, $H_{3'}$, $H_{5'}$), 7.90-7.86 (4H, m, H_4, $H_{4''}$), 7.74-7.69 (8H, m, $H_{06\text{-}09}$, $H_{06'\text{-}09'}$), 7.39 (2H, d, $H_{2'''}$, $H_{6'''}$), 7.36 (2H, m, H_5, $H_{5''}$), 7.17 (2H, d, $H_{3'''}$, $H_{5'''}$), 3.79 (4H, m, H_{02}, $H_{02'}$), 3.69 (2H, s, H_7), 2.84 (4H, m, H_{01}, $H_{01'}$). ^{13}C NMR (75 MHz; solvent $CDCl_3$): δ 168.20 (4C, C_{04}), 156.27, 155.72, 149.76, 149.06 (2C, C_6, $C_{6''}$), 140.12, 136.81 (2C, C_4, $C_{4''}$), 136.59, 133.71 (4C, aromatic phthalimide), 132.31 (4C), 129.57 (2C, $C_{3'''}$, $C_{5'''}$), 126.76 (2C, $C_{2'''}$, $C_{6'''}$), 123.77 (2C, C_5, $C_{5''}$), 123.00 (4C, aromatic phthalimide), 121.27 (2C, C_3, $C_{3''}$), 118.64 (2C, $C_{3'}$, $C_{5'}$), 57.76 (C_7), 51.91 (2C, C_{01}, $C_{01'}$), 35.71 (2C, C_{02}, $C_{02'}$). IR (KBr): $\tilde{\nu}$/cm^{-1} = 3051 w, 3016 w, 2943 w, 2822 m, 1773 s, 1711 ssh, 1603 m, 1583 s, 1568 m, 1542 m, 1514 w, 1468 m, 1431 m, 1396 ssh, 1327 m, 1265 w, 1190 w, 1170 w, 1159 w, 1086 s, 1038 m, 1018 w, 991 w, 966 w,

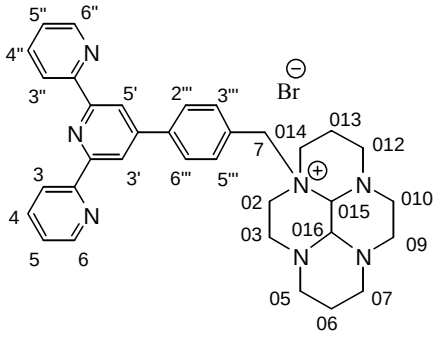
895 w, 872 w, 831 w, 791 s, 746 m, 719 ssh, 687 w, 660 m, 621 m, 530 m, 478 w, 469 w, 415 w. ESI-MS: *m/z* 684.9885 ($[M+H]^+$, 100%).

4'-(*p*-(1,5-bis(amino)-3-azapentane)methylphenyl)-2,2':6',2''-terpyridine (dint)**, 5.33**



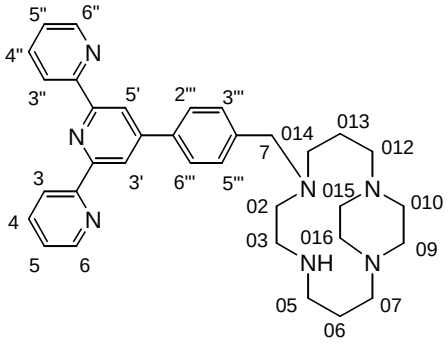
Compound bptt, **5.32,** (0.5 g, 0.73 mmol) in CH_2Cl_2 (25 mL) was added into a MeOH solution of en (0.5 M, 25 mL), and the mixture was stirred for 48 hr at room temperature, filtered, and the filtrate was concentrated to dryness to give a yellow oil material. The mixture was dissolved into $CHCl_3$ (50 mL). The $CHCl_3$ solution was washed with 3% NH_4OH solution (3 × 50 mL), and the organic layer was dried over $MgSO_4$, filtered, the filtrate was concentrated to dryness to give a yellow non-solid material in a quantitative yield. ^1H NMR (500 MHz; solvent $CDCl_3$): δ 8.725 (2H, s, $H_{3'}$, $H_{5'}$), 8.72 (2H, d, H_6, $H_{6''}$), 8.67 (2H, d, H_3, $H_{3''}$), 7.88-7.83 (4H, m, H_4, $H_{4''}$, $H_{2'''}$, $H_{6'''}$), 7.69 (2H, m, H_{06}, $H_{06'}$, 7.44 (2H, d, $H_{3'''}$, $H_{5'''}$), 7.34 (2H, m, H_5, $H_{5''}$), 3.66 (2H, s, H_7), 2.79 (4H, m, H_{02}, $H_{02'}$), 2.56 (4H, m, H_{01}, $H_{01'}$), 1.92 (4H, b, NH_2). ^{13}C NMR (75 MHz; solvent $CDCl_3$): δ 156.17 (2C), 155.84 (2C), 149.94, 149.06 (2C, C_6, $C_{6''}$), 140.55, 137.19, 136.81 (2C, C_4, $C_{4''}$), 129.35 (2C, $C_{3'''}$, $C_{5'''}$), 127.23 (2C, $C_{2'''}$, $C_{6'''}$), 123.76 (2C, C_5, $C_{5''}$), 121.29 (2C, C_3, $C_{3''}$), 118.73 (2C, $C_{3'}$, $C_{5'}$), 58.92 (C_7), 57.08 (2C, C_{01}, $C_{01'}$), 39.66 (2C, C_{02}, $C_{02'}$). IR (KBr): $\tilde{\nu}/cm^{-1}$ = 2802 brs,1653 m, 1585 s, 1568 m, 1541 m, 1516 m, 1470 shs, 1443 w, 1418 w, 1391 m, 1315 w, 1265 w, 1115 brs, 1040 w, 1016 w, 991, w, 895 m, 831 w, 791 shs, 741 m, 719 w, 687 m, 617 m, 579 w, 525 w, 509 w,486 w, 469 w, 459 w, 440 w, 419 m. ESI-MS: *m/z* 213.1583 ($[M+2H]^{2+}$, 100%), 425.3123 ($[M+H]^+$, 9%).

4'-(*p*-(10bα,10cα)-decahydro-1*H*,6*H*-3a,5a,8a,10a-tetraazapyrenium-3a-methylphenyl)-2,2':6',2"-terpyridine bromide (ptmtb), **5.34**



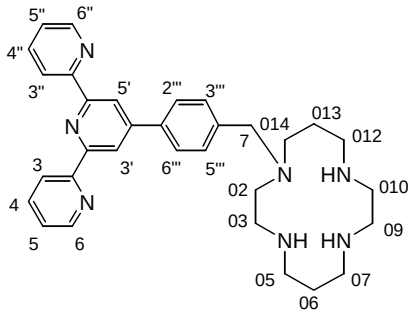
A solution of (10bα,10cα)-decahydro-1*H*,6*H*-3a,5a,8a,10a-tetraazapyrene (capped-cyclam), **5.10**, (0.222 g, 1.0 mmol) and 4'-(*p*-bromomethylphenyl)-2,2':6',2"-terpyridine (btp), **1.50**, (0.40 g, 1.0 mmol), in dry THF (35 mL) was stirred at room temperature for 4 days. The resulting white precipitate was filtered off, washed with diethyl ether, and air-dried to afford an analytically pure powder. The volume of the filtrate was reduced to *ac.* 10 mL. The precipitate was separated by filtration, washed with diethyl ether to obtain the second crop. Yield (total) 11%. ^{1}H NMR (500 MHz; solvent D_2O): δ 7.87 (2H, d, H_6, $H_{6"}$), 7.28 (2H, d, H_3, $H_{3"}$), 7.25-7.22 (2H, m, H_4, $H_{4"}$), 7.00 (2H, s, $H_{3'}$, $H_{5'}$), 6.94-6.92 (2H, m, H_5, $H_{5"}$), 6.87 (2H, d, $H_{3'''}$, $H_{5'''}$), 6.79 (2H, d, $H_{2'''}$, $H_{6'''}$), 4.69-4.66 and 4.19-4.16 (AB, 2H, H_{7A}, H_{7B}), 4.05 (1H, br s, H_{015}), 3.88-3.67 (1H, $H_{02(ax)}$), 3.50 (1H, br s, H_{016}), 3.36-3.34 (1H), 3.23 (1H, $H_{014(ax)}$), 2.94-2.88 (6H, including $H_{03(eq)}$, $H_{05(ax)}$, $H_{012(eq)}$), 2.76-2.74 (2H, including $H_{014(eq)}$), 2.53-2.50 (2H, $H_{02(eq)}$, $H_{03(ax)}$), 2.37-2.35 (2H, including $H_{05(eq)}$), 2.15-2.13 (2H, $H_{012(ax)}$, $H_{06(eq)}$), 1.95-1.93 (1H, $H_{013(eq)}$), 1.60-1.58 (1H, $H_{013(ax)}$), 1.37-1.35 (1H, $H_{06(ax)}$). ^{13}C NMR (75 MHz; solvent D_2O): δ 153.71, 153.30, 147.90 (2C, C_6, $C_{6"}$), 146.26, 137.79, 137.54, 133.21 (2C, $C_{3'''}$, $C_{5'''}$), 126.47, 125.93 (2C, $C_{2'''}$, $C_{6'''}$), 124.29, 121.44 (2C, C_3, $C_{3"}$), 116.72 (2C, $C_{3'}$, $C_{5'}$), 82.62 (C_{016}), 69.50 (C_{015}), 61.39 (C_7), 59.84 (C_{014}), 53.98 (C_{03}), 53.29 (C_{05}), 51.91 (C_{012}), 51.35 , 47.86 (C_{02}), 46.52, 41.90, 18.40 (C_{013}), 17.95 (C_{06}). ESI-MS: *m/z* 272.6939 ($[M+H-Br]^{2+}$, 100%), 544.3924 ($[M-Br]^{+}$, 10%).

4'-(*p*-(10bα,10cα)-decahydro-1*H*,6*H*-3a,5a,8a,10a-tetraazapyrenium-3a-methylphenyl)-2,2':6',2''-terpyridine (pcymt), **5.35**



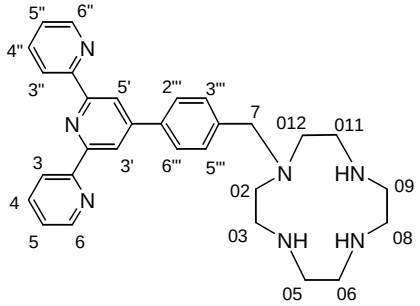
A modified method was adopted for ring cleavage reduction of ptmtb, **5.34**.[295] To a stirred solution of **5.34** (0.5 g) in 95% EtOH (40 mL) was added excess $NaBH_4$ in small portions over 1 hr. The reaction micture was stirred at r.t for 16 days. Excess $NaBH_4$ was then decomposed by slow addition of 3M HCl at 0ºC, and the solvent was removed on vacuum.The resulting white solid was dissolved in water, the pH was adjusted to 14 (by adding KOH conc solution with cooling), and the basic solution was extracted with bezene, then CH_2Cl_2. The combined extracts were dried using $MgSO_4$, filtered, and the solvent was removed to afford a white solid. Yield 85%. ^{1}H NMR (500 MHz; solvent $CDCl_3$): 8.65 (2H, s, $H_{3'}$, $H_{5'}$), δ 8.62 (2H, d), 8.59 (2H, d), 7.80-7.78 (4H, m, including $H_{2'''}$, $H_{6'''}$), 7.32 (2H, d, $H_{3'''}$, $H_{5'''}$), 7.28 (2H, m), 3.59 (2H, s, H_7), 3.26-3.24 (2H, m, H_{010} or H_{015}), 3.08 (2H, m, H_{010} or H_{015}), 2.80-2.76 (2H, t, H_{012}), 2.69 (4H, br, H_{02} or H_{05}), 2.60 (2H, H_{07}), 2.54 (4H, m, H_{03} or H_{014}), 2.48-2.44 (2H, m, H_{09} or H_{016}), 2.26 (2H, m, H_{09} or H_{016}), 1.88 (2H, m, H_{06}), 1.67 (2H, m, H_{013}). ^{13}C NMR (75 MHz; solvent $CDCl_3$): δ 155.96, 149.49, 148.99, 138.79, 137.60, 136.88 (2C, $C_{2'''}$, $C_{6'''}$), 129.81 (2C, $C_{3'''}$, $C_{5'''}$), 128.20, 127.24, 123.56, 121.31, 118.58 (2C, $C_{3'}$, $C_{5'}$), 57.59 (C_7), 56.09 (C_{07}), 55.59 (C_{012}), 53.55 (C_{02}), 50.63 (2C, C_{09}, C_{016}), 50.14 (C_{05}), 48.69 (2C, C_{010}, C_{015}), 47.14 (C_{03}), 23.39 (C_{06}), 23.13 (C_{013}).

4'-(*p*-(1,4,8,11-tetraazacyclotetradec-1-yl)methylphenyl)-2,2':6',2''-terpyridine (cymt), **1.74**



A solution of 4'-(*p*-bromomethylphenyl)-2,2':6',2''-terpyridine (btp), **1.50**, (0.40 g, 1.0 mmol), in dry toluene (20 mL) under Ar was added dropwise to a stirred mixture of 1,4,8,11-tetraazacyclotetradecane (cyclam)[459] (1.0 g, 5 mmol) and anhydrous K_2CO_3 (3.5 g, 25 mmol) in dry toluene at 30 ºC over a period of 2 hr. The reaction mixture was stirred at 100 ºC overnight, cooled at r.t, filtered to remove K_2CO_3 and excess cyclam (Excess cyclam was then recovered by washing the solid residual well with CH_3Cl, and removal of the solevent on a rotary evaporator). Toluene was taken off the filtrate on vacuum to afford a pale oil which was solidified on scraching. The crude material was purified by column chromatography (Al_2O_3, eluting with CH_2Cl_2 then with 2-3% MeOH in CH_2Cl_2) to afford a glassy off-white solid. Yield 0.277 g, 53%. ^{1}H NMR (500 MHz; solvent $CDCl_3$): δ 8.74-8.72 (2H,d), 8.735 (2H, s, $H_{5'}$, $H_{3'}$), 8.68 (2H, d), 7.90-7.87 (4H, m), 7.50 (2H, d, $H_{5'''}$, $H_{3'''}$), 7.37-7.35 (2H, m), 3.65 (2H, s, H_7), 2.88 (2H, m), 2.84 (2H, m), 2.77-2.72 (4H, m), 2.68-2.59 (4H, m), 2.57 (2H, m), 2.56 (2H, m), 1.90-1.88 (2H, m, H_{013}), 1.73-1.71 (2H, m, H_{06}). ^{13}C NMR (75 MHz; solvent $CDCl_3$): δ 156.31, 155.91, 150.06, 149.12 (2C), 140.06, 137.11, 136.87 (2C), 129.70 (2C), 127.13 (2C), 123.80 (2C), 121.39 (2C), 118.74 (2C), 57.73, 54.47, 53.39, 50.74, 49.32, 49.14, 49.00, 47.91, 47.43, 28.40, 26.18. ESI-MS: *m/z* 522.3300 ($[M+H]^+$, 100%).

4'-(*p*-(1,4,7,10-tetraazacyclododec-1-yl)methylphenyl)-2,2':6',2''-terpyridine (cynt), **1.75**



A modified literature procedure[197] was adopted for preparation of this ligand. A solution of 4'-(*p*-bromomethylphenyl)-2,2':6',2''-terpyridine (btp), **1.50**, (0.40 g, 1.0 mmol), in dry CH_2Cl_2 (20 mL) under Ar was added dropwise to a stirred solution of 1,4,7,10-tetraazacyclododecane[462,463] (cyclen) (0.86 g, 5 mmol) in dry CH_2Cl_2 (20 mL). Et_3N (5 drops) was added, and the mixture was heated at 30 ºC for 24 hr. The reaction mixture was cooled at r.t and washed with water (3 × 10 mL). The aqueous layer was kept aside to recover the excess cyclen. The organic phase was collected, dried with $MgSO_4$, filtered, and the solvent was removed by rotary evaporation. The crude material was purified by column chromatography (Al_2O_3, eluting with 1-2% MeOH in CH_2Cl_2) to afford a glassy white solid. Yield 0.237 g, 48%. . ^{1}H NMR (300 MHz; solvent $CDCl_3$): δ 8.74 (2H, s, $H_{5'}$, $H_{3'}$), 8.72 (2H,d), 8.68 (2H, d), 7.91-7.85 (4H, m), 7.47 (2H, d), 7.37-7.35 (2H, m), 3.70 (2H, s, H_{01}), 2.85-2.62 (16H, m). ^{13}C NMR (75 MHz; solvent $CDCl_3$): δ 156.20, 155.79, 149.88, 149.04 (2C), 140.11, 137.07, 136.77 (2C), 129.57 (2C), 127.22 (2C), 123.70 (2C), 121.27 (2C), 118.68 (2C), 58.79, 51.22, 46.94, 46.16, 29.62. ESI-MS: *m/z* 494.2987 ($[M+H]^+$, 100%).

8.3. Syntheses of Complexes

[Ni(tpt)(H_2O)$_3$)]Cl_2.2H_2O, **2.7**, and {[Ni(tpt)Cl(H_2O)$_2$]Cl, **2.8,** + [Ni(tpt)Cl_2(H_2O)], **2.9**}.4H_2O. (*Method a*)

To a solution of tpt, **1.70**, (0.312 g, 1 mmol) in MeOH (10 mL) was added $NiCl_2.6H_2O$ (0.2379 g, 1 mmol) in water (10 mL), with stirring. The green reaction mixture was heated at reflux for 1hr, cooled to room temperature and then the volume of the solution was reduced to *ca*. 5 mL on a rotary evaporator. Vapour diffusion of acetone into the aqueous solution resulted in the formation of large blocks of green crystalline material **2.7** over a few days as the major product, followed by formation of orange needle-like crystals over a week (**2.8**) + (**2.9**). Crystalline materials of **2.7** and (**2.8**) + (**2.9**) were used for the analyses and structural determinations. IR (KBr) for compound (**2.7**): $\tilde{\nu}$/cm^{-1} = 3263 mb, 3188 m, 3136 m, 3117m, 1607 w, 1562 s, 1537 s, 1493 m, 1475 w, 1448 w, 1410 m, 1391 m, 1379 s, 1308 w, 1263 m, 1184 w, 1155 w, 1096 m, 1057 w, 1036 m, 1024 m, 1013 m, 870 m, 849 m, 808 m, 775 s, 760 m, 687 m, 665 m, 638 m, 525 w. Found: C 42.33, H 3.80, N 16.40%. Calc. for $C_{18}H_{18}Cl_2N_6NiO_3.H_2O$, **2.7**: C 42.06, H 3.92, N 16.35%. IR (KBr) for compounds (**2.8**) + (**2.9**): $\tilde{\nu}$/cm^{-1} = 3250 mb, 3120 m, 1603 w, 1574 s, 1553 s, 1483 m, 1389 s, 1342 m, 1306 m, 1269 m, 1279 w, 1219 m, 1184 m, 1163 w, 1097 m, 1045 m, 1030 w, 1011 s, 860 m, 775 s, 679 m, 667 m, 637 w, 617 w.

{[Ni(tpt)Cl(H_2O)$_2$]Cl, **2.8** + [Ni(tpt)Cl_2(H_2O)]}.4H_2O, **2.9**. (*Method b*)

Green crystals of compound **2.7** (0.085 g, 0.165 mmol) were dissolved in water (5 mL). To this solution was added excess NaCl, with stirring, until a saturated solution was obtained. The green-orange solution was filtered off to remove undissolved NaCl. Vapour diffusion of acetone into the aqueous solution over a week

resulted in the formation of rods of orange crystals suitable for X-ray diffraction. The Infrared spectra and the crystallographic data for this product were identical to those for compound obtained by *Method a*.

[Ni(HTPT)Cl(H_2O)$_2$]Cl_2.2H_2O, **2.10a** and **2.10b**

Green crystals of compound **2.7** (0.075 g, 0.146 mmol) were dissolved in water (5 mL). To this green solution was added conc. HCl (1 mL) drop-wise to obtain a green-orange solution, then concentrated to *ca*. 2 mL on a rotary evaporator. Orange needle-like crystals suitable for X-ray diffraction were obtained by vapour diffusion of acetone into the aqueous solution over a week (**2.10a**). IR (KBr): $\tilde{\nu}$/cm^{-1} = 3300 mb, 3096 w, 3059 w, 1603 w, 1574 s, 1553 s, 1483 m, 1452 w, 1398 s, 1389 s, 1342 w, 1306 m, 1279 w, 1269 w, 1263 w, 1219 m, 1184 w, 1163 w, 1097 m, 1045 m, 1030 w, 1011 s, 980 w, 860 m, 775 s, 737 w, 679 w, 667 m, 637 w, 617 w, 544 w, 532 w, 517 w. Found: C 38.34, H 3.81, N 14.85%. Calc. for $C_{18}H_{17}Cl_3N_6NiO_2$.2.5H_2O: C 38.64, H 3.96, N 15.02%.

Evaporation of acetone from the solution containing the crystalline materials yielded to a clear solution which afforded a hexagonal prism **2.10b** suitable for X-ray diffraction upon evaporation of the aqueous media. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3300 mb, 3215 mb, 3096 m, 3059 m, 3003 m, 2822 m, 2760 m, 2586 w, 2355 w, 2332 w, 2137 w, 2035 w, 2019 w, 1977 w, 1963 w, 1919 w,1900 w, 1854 w, 1805 w, 1607 m, 1576 s, 1553 s, 1481 m, 1450 w, 1400 s, 1389 s, 1364 w, 1340 w, 1308 m, 1292 w, 1279 w, 1258 w, 1234 m, 1184 w, 1163 w, 1151 w, 1109 w, 1096 m, 1045 m, 1030 m, 1013 s, 959 w, 922 w, 860 m, 777 s, 737 w, 679 m, 667 m, 638 m, 617 m, 546 w, 513 w.

$[Ni(tpt)(H_2O)_3].(NO_3)_2$, **2.12.**

To a solution of tpt, **1.70** (0.312 g, 1 mmol) in MeOH (10 mL) was added $Ni(NO_3)_2.6H_2O$ (0.291 g, 1 mmol) in water (2 mL). The resulting orange-green solution was allowed to reflux for 1 hr, then cooled at room temperature, concentrated to *ca*. 5 mL on a rotary evaporator. Acetone vapour diffusion into the aqueous solution of the complex over two weeks resulted in the formation of blocks of green crystals suitable for X-ray crystallography. The crystals were collected by filtration, washed with acetone, then with diethyl ether, and air-dried. Yield 0.32 g. IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3506 m, 3431 m, 3186 mb, 3144 mb, 3069 m, 3022 m, 1749 w, 1684 m, 1653 m, 1611 m, 1576 s, 1560 s, 1541 s, 1489 m, 1475 m, 1385 s, 1323 s, 1259 m, 1186 m, 1157 m, 1115 w, 1094 m, 1045 m, 1015 m, 976 w, 860 m, 826 m, 773 s, 758 m, 685 m, 667 m, 638 m, 627 m, 592 w, 538 w, 511 w. Found: C 38.82, H 3.57, N 20.08%. Calc. for $C_{18}H_{18}N_8NiO_9.0.5H_2O$: C 38.74, H 3.43, N 20.08%. ES^+ MS *m/z* calcd for $[Ni(tpt)(H_2O)_3](NO_3)^+$ 744; found 744.

$[Ni(tpt)_2](ClO_4)_2$, **2.13.**

Method (a): To a solution of **1.70** (0.312 g, 1 mmol) in MeOH was added $Ni(ClO_4)_2.6H_2O$ (0.366 g, 1 mmol) in water with stirring. A brown precipitate was formed immediately. Stirring was continued at reflux for 30 min, and the solution was allowed to stand at room temperature for two hours. The brown precipitate was separated from a green solution by filtration, washed well with MeOH, diethyl ether, and air-dried to give a light brown powder. Yield 0.32 g (73%). IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3516 wb, 3277 w, 3057 w, 3011 w, 2936 w, 2023 w, 1692 w, 1659 w, 1649 w, 1611 m, 1580 s, 1562 s, 1533 s, 1487 m, 1450 m, 1439 m, 1398 s, 1379 s, 1306 m, 1256 m, 1182 m, 1157 m, 1099 s, 1049 m, 1030 m, 1013 m, 995 m, 980 w, 957 w, 924 w, 858

m, 845 w, 816 m, 772 s, 739 m, 687 m, 665 m, 638 m, 623 s, 546 w, 527 w, 511 w. ESI-MS *m/z* calcd for $\{[Ni(tpt)_2](ClO_4)\}^+$ 781 ;found 781. Found: C 48.08, H 2.78, N 18.96%. Calc. for $C_{36}H_{24}Cl_2N_{12}NiO_8.H_2O$: C 48.03, H 2.91, N 18.67%. Crystals suitable for X-ray diffraction were obtained by vapour diffusion of MeOH into DMSO solution of the complex.

Method (b): To a green solution of **2.7** (0.248 g, 0.5 mmol) in water was added excess $NaClO_4$ with stirring. A brown precipitate which was formed immmediately was separated from a weakly coloured solution by filtration, washed well with water, then with diethyl ether, and air-dried to afford a light brown powder. Yield 0.2 g, 90%. Infrared spectra of this product was identical to that of compound **2.133** obtained by method described as above.

$[Ni(tpt)_2](Cl)_2.nH_2O$, **2.14.**

To a stirring solution of **1.70** (0.312 g, 1 mmol) in MeOH (10 mL) was added $NiCl_2.6H_2O$ (0.119 g, 0.5 mmol) in water. The orange-red solution was allowed to reflux for 1 hr, and then cooled at room temperature. The resultant brown solution was concentrated in volume on a rotary evaporator. Slow evaporation of the solution in the refrigerator afforded brown-red crystals suitable for X-ray diffraction. IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3344 bs, 3194 bs, 3065 w, 2887 w, 2772 w, 2029 w, 1888 w, 1665 w, 1611 m, 1560 m, 1533 s, 1487 m, 1439 m, 1396 s, 1304 w, 1258 s, 1184 w, 1159 m, 1094 m, 1049 m, 1017 s, 999 w, 914 w, 858 s, 812 w, 772 s, 685 m, 665 m, 638 m, 627 w, 525 w, 503 w.

$[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15.**

To a stirring solution of tpt, **1.70**, (0.312 g, 1 mmol) in EtOH (10 mL) was added excess $Ni(NO_3)_2.6H_2O$ (2.907 g, 10 mmol) in EtOH (5 mL). The green solution was heated on the steam bath until a green crystalline material was precipitated. The green crystals were separated from the hot green solution, washed with ethanol, acetone, then air-dried to give green needles suitable for X-ray diffraction crystallography. Yield 0.42 g. IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3065 sb, 2988 w, 1767 w, 1541 ssh, 1522 msh, 1385 msh, 1300 msh, 1265 msh, 1184 w, 1163 w, 1040 ssh, 1016 ssh, 874 w, 826 w, 802 w, 772 ssh, 756 w, 669 w, 640 w, 486 w, 426 msh. Found: C 33.19, H 3.34, N 17.79%. Calc. for $C_{22}H_{26}N_{10}Ni_2O_{15}$: C 33.54, H 3.33, N 17.78. ESI-MS *m/z* calcd for $[Ni_2(tpt)_2(NO_3)_3]^+$ 926; found 926. calcd for $[Ni(tpt)_2](NO_3)^+$ 744; found 744.

$[Ni_2(tpt)Cl_2(H_2O)_5]Cl_2.nH_2O$ (**2.16**) + (**2.17**).

To a stirring solution of **1.70** (0.312 g, 1 mmol) in MeOH (10 mL) was added excess $NiCl_2.6H_2O$ (2.38 g, 10 mmol) in MeOH (10 mL). The orange-green solution was heated to reflux for 30 min. The solution was reduced in volume on a steam bath until a yellow precipitate was formed. The precipitate was separated by filtration while still hot, washed with small portion of MeOH, diethyl ethyl ether, and air-dried to give an orange powder. Yield 0.2 g, 37%. IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3072 sb, 2972 w, 1541 ssh, 1491 msh, 1473 msh, 1448 w, 1412 w, 1379 msh, 1304 w, 1265 msh, 1161 w, 1099 w, 1081 w, 1038 w, 1013 msh, 870 w, 773 ssh, 685 w, 669 w, 638 w, 455 w, 428 w, 419 w. Found: C 35.21, H 3.01, N 13.14%. Calc. for $C_{18}H_{18}C_{l4}N_6Ni_2O_3$: C 34.56, H 2.90, N 13.43%.

[Ni(tpt)$(MeOH)_2$Cl]Cl, **2.18**.

Vapour diffusion of acetone into the methanolic solution of complexes (**2.16**) + (**2.17**) afforded green needle-like crystals suitable for X-ray diffraction crystallography. Yield 86%. IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3572 mb, 3140 sb, 1543 ssh, 1489 w, 1443 w, 1381 ssh, 1257 msh, 1188 w, 1096 w, 1011 m, 856 w, 772 ssh, 687 w, 640 w, 486 w, 424 w.

[Ru$(tpm)_2$Cl]Cl, **3.4**.

To [Ru(tpm)Cl_3], **3.2**, (0.300 g, 0.712 mmol) and LiCl (0.300 g) in water: EtOH (1: 3) (40 mL) was added tpm ligand, **3.1**, (0.152 g, 0.712 mmol). The reaction mixture was heated at reflux for 5 min. To the dark green-brown solution was added Et_3N (12 drops) before it was refluxed for further 10 min to give blue-green solution. The volume of the mixture was reduced to *ca*. 20 mL on vacuum after it was cooled at r.t. The mixture was kept in the refrigerator overnight. A green precipitate which was formed was separated from a blue solution by filtration, washed with cold water, then air-dried to afford a green powder. Yield 0.3 g, 57%. ^{1}H NMR (500 MHz; solvent dmso-d_6) δ 10.58 (1H,s), 10.28 (1H, s), 8.78 (2H, d), 8.74 (1H, d), 8.71 (1H, d), 8.34 (1H, d), 8.04 (2H, d), 7.67 (2H, d), 7.25 (2H, d), 6.94 (1H, dd), 6.83 (2H, dd), 6.66 (2H, dd), 6.62 (1H, d), 6.56 (1H, dd). ^{13}C NMR (75 MHz; solvent dmso-d_6) δ 147.09 (2C), 146.14 (2C), 145.96 (1C), 144.91 (1C), 135.32 (1C), 133.37 (1C), 130.04 (1C), 109.05 (1C), 108.95 (2C), 108.63 (2C), 108.16 (1C), 80.59 (1C), 75.37 (1C). IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3094 m, 1514 w, 1474 m, 1407 s, 1394 m, 1254 m, 1229 w, 1088 s, 1061 m, 1022 w, 989 w, 910 w, 858 m, 833 m, 787 m, 762 s, 610 w, 588 w. ESI-MS: *m/z* 565.2 ([M-Cl]$^+$), 265.03 ([M-Cl]$^{2+}$). UV-vis (CH_3CN): λ_{max} (ε) = 230.0 (9300), 280.0 (4900), 290.0 (4900), 335.0 (7300) nm (L mol^{-1} cm^{-1}). Anal. Calc. for

$C_{20}H_{20}Cl_2N_{12}Ru.1.5H_2O$ (627.46): C 38.28, H 3.69, N 26.79%; found: C 38.24, H 3.43, N 26.45.

[Ru(tpm)$_2$(Cl)](ClO$_4$), **3.5**.

[Ru(tpm)$_2$Cl]Cl, **3.4**, (0.15 g, 0.25 mmol) and $AgClO_4.H_2O$ (0.112 g, 0.5 mmol) in 30 mL of acetone were heated at reflux for 4 h. AgCl precipitate was filtered off and the blue solution was taken to dryness in a rotary evaporator. Slow evaporation of an acetone-benzene solution of the mixture afforded some pale green crystals with poor quality. X-ray diffraction revealed that compound **3.6** was formed as a side product. The bulk blue solution was separated from the pale crystals through filtration. The product was recrystallized form acetone-benzene, washed with diethyl ether, and air-dried to afford a blue powder. Yield 0.17 g, 85%. ^{1}H NMR (300 MHz; solvent dmso-d_6) δ 10.03 (1H, s), 8.89 (1H, s), 8.78 (3H, m), 8.66 (1H, d), 8.38 (1H, d), 8.15 (2H, d), 7.70 (2H, d), 7.37 (2H, d), 6.99 (1H, t), 6.94 (2H, t), 6.74 (2H, dd), 6.68 (1H, dd), 6.56 (1H, d), 6.42 (2H, b s). IR (KBr): $\tilde{\nu}$/cm^{-1} = 3140 b, 3042 w, 1628 w, 1520 w, 1441 m, 1414 m, 1377 w, 1310 m, 1285 msh, 1252 m, 1231 w, 1094 ssh, 1061 m, 991 w, 962 w, 845 s, 756 s, 606 m, 559 ssh. ESI-MS: *m/z* 629.09 ([M-Cl]$^+$), 565.11 ([M-ClO$_4$]$^+$), 265.06 ([M-Cl-ClO$_4$]$^{2+}$).

[Ru(tpm)$_2$(H$_2$O)](PF$_6$)$_2$, **3.7**.

[Ru(tpm)$_2$Cl]Cl, **3.4**, (0.15 g, 0.25 mmol) and $AgClO_4.H_2O$ (0.112 g, 0.5 mmol) in 30 mL of acetone: water (3: 1) were heated at reflux for 2 h. The pot content was chilled in a refrigerator for 2 h, after AgCl precipitate was filtered off. To the cold solution was added excess NH_4PF_4. The blue precipitate was collected by filtration through Celite, dissolved in CH_3CN and was purified by column chromatography

(silica gel eluting with CH_3CN/saturated aqueous KNO_3/water (17:0.5:1)). An excess of NH_4PF_6 was added to the major blue fraction and the solution reduced in volume. The precipitate was collected by filtration through Celite, dissolved in CH_3CN and evaporated to dryness to give $[Ru(tpm)_2(H_2O)](PF_6)_2$ as a blue powder. Further purification was achieved by recrystallisation from CH_3CN-H_2O solution of the complex. Yield 0.25 g, 90 %. ^{1}H NMR (500 MHz; solvent dmso-d_6) δ 10.03 (1H, s), 8.89 (1H, s), 8.77 (2H, d), 8.75 (1H, d), 8.63 (1H, d), 8.36 (1H, d), 8.14 (2H, d), 7.68 (2H, d), 7.36 (2H, d), 6.97 (1H, d), 6.92 (2H, t), 6.71 (2H, t), 6.65 (1H, dd), 6.54 (1H, t), 6.45 (2H(coordinated water molecule), b s). ^{13}C NMR (75 MHz; solvent dmso-d_6) δ 147.72 (1C), 147.55 (2C), 147.40 (2C), 145.53 (1C), 136.62 (1C), 136.07 (2C), 135.97 (1C), 110.39 (1C), 109.83 (1C), 109.64 (2C), 109.63 (2C), 81.85 (1C), 76.49 (1C). IR (KBr): $\tilde{\nu}$/cm^{-1} = 3140 b, 3042 w, 1628 w, 1520 w, 1441 m, 1414 m, 1377 m, 1285 m, 1252 w, 1231 w, 1094 s, 1061 m, 991 w, 962 w, 845 ssh, 756 s, 606 w, 559 s. ESI-MS: *m/z* 693.07 ($[M\text{-}PF_6]^+$), 274.05 ($[M\text{-}2PF_6]^{2+}$). UV-vis (CH_3CN): λ_{max} 305.0, 590.0 nm.

$[Ru(tpm)_2(CH_3CN)](ClO_4)_2$, **3.8**.

Method 1: $[Ru(tpm)_2Cl]Cl$, **3.4**, (0.15 g, 0.25 mmol) and $AgClO_4.H_2O$ (0.112 g, 0.5 mmol) in 30 mL of dry acetonitrile were heated at reflux for 4 h. AgCl was filtered off and the yellowish solution was taken to dryness under vacuum. The crude material was recrystallized by vapour diffusion of diethyl ether into the acetonitrile solution of the complex. Yield 0.11 g, 58%. ^{1}H NMR (300 MHz; solvent dmso-d_6) δ 10.06 (1H, s), 8.96 (1H, s), 8.77 (2H, d), 8.75 (1H, d), 8.69 (1H, d), 8.37 (1H, d), 8.16 (2H, d), 7.68 (2H, d), 7.34 (2H, d), 6.91-6.94 (3H, m), 6.70-6.71 (3H, m), 661 (1H, dd), 2.7 (3H (coordinated CH_3CN), s). ^{13}C NMR (75 MHz; solvent dmso-d_6) δ 149.86,

147.11, 146.65, 145.79, 145.14, 141.20,136.76, 135.90, 134.68, 130.42, 130.20, 124.27, 118.21, 109.58, 109.24, 108.84, 108.14, 107.06, 80.75, 75.79, 1.26. IR (KBr): $\tilde{\upsilon}$/cm^{-1} = 3123 msh, 2995 msh, 1518 m, 1472 w, 1441 m, 1412 ssh, 1379 w, 1286 m, 1250 m, 1090 ssh, 991 w, 955 w, 862 m, 837 m, 818 m, 758 s, 625 ssh, 608 m, 446 w. ESI-MS: *m/z* 670.13 ([M-ClO_4]$^+$), 285.59 ([M-2ClO_4]$^{2+}$). UV-vis (CH_3CN): λ_{max} 265.1, 290.0, 305.0 nm.

Method 2: [Ru(tpm)$_2$(H_2O)](PF_6)$_2$, **3.7**, and acetonitrile (2 mL) in 20 mL acetone: water (3:1) were heated at reflux for 24 h. The yellow-orange solution was taken to dryness after it was cooled at r.t. The crude material was recrystallised by vapour diffusion of diethyl ether into the acetonitrile solution of the complex. The precipitate was separated by filtration, washed with ether, then air-dried to give a yellow-orange powder. Yield 0.14 g, 74%.

[Ru(tpm)(bpy)Cl](PF_6), **3.10**.

X-ray quality crystals were obtained by vapour diffusion of diethyl ether into a MeOH solution of the complex in seven days. ^{1}H NMR (300 MHz; solvent dmso-d_6) δ 10.24(s, 1H), 8.87 (d, 2H), 8.76-8.75 (m, 4H), 8.62 (d, 1H), 8.38 (d, 2H), 8.19 (dd, 2H), 7.65 (dd, 2H), 6.89 (m, 2H), 6.81 (d, 1H), 6.45 (m, 1H). ^{13}C NMR (75 MHz; solvent dmso-d_6) δ 158.88, 152.29, 147.45, 144.62, 136.07, 135.58, 134.49, 125.76, 123.52, 109.16, 108.71, 75.30.

[Ru(tpm)(bpy)(OH_2)](ClO_4)$_2$, **3.11**.

X-ray quality crystals were obtained by vapour diffusion of diethyl ether into a MeOH solution of the complex in two days. ^{1}H NMR (300 MHz; solvent dmso-d_6) δ 9.26 (s, 1H), 8.95 (d, 2H), 8.77-8.76 (m, 4H), 8.56 (d, 1H), 8.49 (d, 2H), 8.30 (dd, 2H),

7.75 (dd, 2H), 6.98 (m, 2H), 6.88 (d, 1H), 6.45 (m, 1H), 5.76 (s, 2H, coordinated H_2O molecule). ^{13}C NMR (75 MHz; solvent dmso-d_6) δ 158.90, 153.15, 147.43, 146.02, 137.32, 136.89, 135.68, 126.32, 123.95, 109.44, 108.19, 75.63.

[Ru(ttp)(bpy)Cl](PF$_6$), **3.13**.

To [Ru(ttp)Cl$_3$], **3.2**, (0.237 g, 0.539 mmol) in 80 mL EtOH:water (4:1), was added bpy (0.094 g, 0.6 mmol). The mixture was allowed to reflux for 5 h, excess solid LiCl was added, and the mixture was heated for an additional 45 min. To the cold reaction micture was added excess aqueous NH_4PF_6 solution, then the volume of the mixture was reduced under vacuum to about 40 mL. The brown precipitate was collected by filtration through Celite, recrystallized from acetone and diethyl ether (1:5) to afford a brown powder. Further purification was achieved by column chromatography (silica gel eluting with CH_3CN/toluene (2:1)). An excess of NH_4PF_6 was added to the major red-purple fraction and the solution reduced in volume. The precipitate was collected by filtration through Celite, dissolved in CH_3CN and evaporated to dryness to give [Ru(ttp)(bpy)Cl](PF$_6$) as a dark red powder. Yield 0.187 g, 55%. Red blocks of crystals suitable for X-ray determination were obtained by vapour diffusion of diethyl ether into CH_3CN solution of the complex within two days. ^1H NMR (500 MHz; solvent dmso-d_6, see Scheme 3 for numbering) δ 10.23 (d, 1H, H_{6B}), 9.24 (s, 2H, $H_{3'}$, $H_{5'}$), 9.02 (m, 3H, H_3, $H_{3''}$, H_{3B}), 8.74 (d, 1H, H_{3A}), 8.46 (t, 1H, H_{4B}), 8.34 (d, 2H, $H_{2'''}$, $H_{6'''}$), 8.18 (t, 1H. H_{5B}), 8.10 (m, 2H, H_4, $H_{4''}$), 7.87 (t, 1H, H_{4A}), 7.74 (d, 2H, H_6, $H_{6''}$), 7.61 (d, 2H, $H_{3'''}$, $H_{5'''}$), 7.53 (d, 1H, H_{6A}), 7.48 (t, 2H, H_5, $H_{5''}$), 7.18 (t, 1H, H_{5A}), 2.57 (s, 3H, H_7). ^{13}C NMR (75 MHz; solvent dmso-d_6) δ 158.74, 158.44, 157.81, 155.83, 152.10, 151.95, 151.82, 145.19, 139.95, 137.04, 136.66, 135.63, 133.48, 130.00, 127.53, 127.48, 127.01, 126.56, 124.15, 123.84,

123.57, 119.67, 21.05. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3070 m,1605 m, 1520 w, 1462 m, 1427 m, 1404 m, 1354 w, 1312 w, 1292 w, 1250 m, 1196 m, 1161 w, 1018 m sh, 968 w, 841 s sh, 791 s, 760 s, 729 m, 656 m, 559 s, 494 w, 459 w, 424 w. ESI-MS: *m/z* 616.29 ([M-PF_6]$^+$). UV-vis (CH_3CN): λ_{max} (ε) = 285.0 (55880), 295.0 (55561), 504.9 (10025) nm (L mol^{-1} cm^{-1}).

[Ru(ttp)(bpy)(bpe)]$(PF_6)_2$, **3.14**.

To [Ru(ttp)(bpy)Cl], **3.13**, (0.050 g, 0.074 mmol) in 25 mL EtOH: water (1: 1), was added excess bpe (0.128 g, 0.7 mmol). The mixture was refluxed for 5 h. The volume of the cold solution was reduced to half by rotary evaporation. To the resulting yellow-red solution was added excess NH_4PF_6. A dark red microcrystalline material was precipitated immediately. The precipitate was collected by filtration through Celite, after it was kept in the refrigerator for 2 h. The product was then purified by column chromatography (silica gel eluting with CH_3CN/saturated solution KNO_3/water (10:2:1). An excess of NH_4PF_6 was added to the last major red band. The precipitate was collected by filtration through Celite, washed with water, ether, then dissolved in CH_3CN and evaporated to dryness to give the pure product as a dark red powder. Yield 0.048 g, 70%. Red blocks of crystals suitable for X-ray crystallography were grown by vapour diffusion of diethyl ether into CH_3CN/MeOH (1:1) solution of the complex over a week. ^{1}H NMR (500 MHz; solvent acetone-d_6, see Scheme 3 for numbering) δ 9.36 (s, 2H, H_3', H_5'), 9.20 (d, 1H, H_{6B}), 9.12 (d, 3H, H_3, H_{3B}), 8.86 (d, 1H, H_{3A}), 8.60-8.57 (m, 1H, H_{4B}), 8.35-8.32 (dd, 2H, H_4), 8.29 (d, 2H, H_6), 8.27-8.25 (m, 4H), 8.25-8.24 (d, 2H, $H_{2'''}$, $H_{6'''}$), 8.16-8.08 (m, 2H, H_{5B}, H_{4B}), 7.92 (d, 1H, H_F), 7.89 (d, 1H, H_{6A}), 7.82 (d, 1H, H_E), 7.74-7.73 (m, 2H, H_5), 7.73-7.72 (m, 2H), 7.63 (d, 2H, $H_{3'''}$, $H_{5'''}$), 7.39-7.36 (m, 1H, H_{5A}), 2.60 (s, 3H, CH_3). ^{13}C NMR (75 MHz; solvent

acetone-d_6) δ 158.79, 158.05, 157.80, 156.68, 153.50, 153.07, 152.19, 151.57, 148.91, 144.83, 144, 57, 141.22, 139.00, 138.15, 137.83, 134.39, 133.60, 131.36, 130.36, 129.12, 128.21, 127.77, 127.22, 125.48, 124.86, 124.27, 124.07, 121.52, 20.65. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3674 m, 3626 w, 3304 w, 3107 w, 1630 m, 1607 m, 1539 m,1506 m, 1470 m, 1447 w, 1427 m, 1406 m, 1352 w, 1315 w, 1290 w, 1205 w, 1163 w, 1022 w, 978 m, 839 s sh, 789 s, 764 s, 739 m, 718 w, 656 w, 619 w, 559 s sh, 509 w, 494 w, 486 w, 474 w, 418 w. ESI-MS: *m/z* 908.43 ([M-PF_6]$^+$), 381.63 ([M-2PF_6]$^{2+}$). UV-vis (CH_3CN): λ_{max} (ε) = 290.0 (157928), 385.0 (10060), 429.9 (14261) nm (L mol^{-1} cm^{-1}).

[$Ag_2(pzt)_2$](BF_4)$_2$, **4.2**.

To the ligand pzt , **1.71**, (0.023 g, 0.04 mmol) in dry CH_2Cl_2 (5 mL) under Ar atmosphere was added $AgBF_4$ (0.008 g, 0.04 mmol) in dry CH_3CN (5 mL) in dark. The reaction mixture was stirred for two hours at r.t, before it was taken to dryness under vacuum. Recrystallisation of the solid material from CH_3CN-diethyl ether afforded a white powder. Slow evaporation of dmso-d_6 solution of the white powder afforded colourless crystals suitable for X-ray crystallography. ^{1}H NMR (500MHz; solvent dmso-d_6) δ 8.73 (4H, s, $H_{3'}$, $H_{5'}$), 8.67 (4H, d, H_3, $H_{3''}$), 8.50 (4H, d, H_6, $H_{6''}$), 8.20 (4H, m, $H_{2'''}$, $H_{6'''}$), 8.17 (4H, d, H_4, $H_{4''}$), 7.81 (6H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.65 (6H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.59 (4H, dd, H_5, $H_{5''}$), 7.51 (4H, d, $H_{3'''}$, $H_{5'''}$), 6.55 (6H, m, H_{04}, $H_{04'}$, $H_{04''}$), 5.19 (4H, s, H_{01}), 4.79 (4H, s, H_7). ^{13}C NMR (75MHz; solvent dmso-d_6) δ 153.20, 152.00, 150.80, 150.65, 141.11, 139.62, 138.96, 135.69, 131.19, 128.30, 127.84, 125.71, 123.85, 120.92, 106.66, 89.40, 72.70, 72.34. ESI-MS: *m/z* 1433.2 ([M-BF_4]$^+$), 1239.3 ([M-Ag-2BF_4]$^+$), 674.1 ([M-2BF_4]$^{2+}$). Anal. Calc. for $C_{66}H_{54}Ag_2B_2F_8N_{18}O_2.H_2O$ (1538.62): C 51.52, H 3.67, N 16.39%; found: C 51.73, H 3.53, N 16.02%.

Ru(pzt)Cl_3, **4.3**.

To 125 mL of absolute ethanol in a 200 mL round-bottom flask was added $RuCl_3.3H_3O$ (0.033 g, 0.124 mmol) and ligand pzt , **1.71**, (0.070 g, 0.124 mmol). The mixture was heated at reflux for about 3 h while vigorous magnetic stirring was maintained. After this time the reaction was cooled to room temperature, and the fine brown powder which had appeared was filtered from the red-brown solution. The product was washed with 3 × 30 mL portions of diethyl ether and air-dried. Yield 0.0724 g , 75 %.

$[Ru(pzt)_2](PF_6)_2$, **4.4**.

Ligand pzt , **1.70**, (0.083 g, 0.147 mmol) and Ru(pzt)Cl_3 complex , **4.3**, (0.072 g, 0.074 mmol) in dry MeOH (50 mL) in the presence of *N*-methylmorpholine (12 drops) were heated at reflux for 4h under Ar. The deep red solution was cooled to room temperature before it was filtered to remove any unreacted materials. The solution was taken to dryness under vacuum and the residue was purified by column chromatography (SiO2 eluting with CH_3CN-saturated KNO_3 solution-H_2O, 10 :2 :1). An excess of NH_4PF_6 was added to the major red-brown fraction and the solution was reduced in volume under vacuum. The precipitate was collected by filtration over Celite, before it was dissolved in CH_3CN, and evaporated to dryness *in vacuo* to afford $[Ru(pzt)_2](PF_6)_2$ as a red-brown powder. Yield 0.095g, 79 %. ^{1}H NMR (500MHz; solvent acetone-d_6) δ 9.55 (4H, s, $H_{3'}$, $H_{5'}$), 9.18 (4H, d, H_3, $H_{3''}$), 8.41 (4H, d, $H_{2'''}$, $H_{6'''}$), 8.23 (4H, m, H_4, $H_{4''}$), 7.93 (4H, d, H_6, $H_{6''}$), 7.79 (6H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.74 (4H, d, $H_{3'''}$, $H_{5'''}$), 7.69 (6H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.47 (4H, m, H_5, $H_{5''}$), 6.55 (6H, t, H_{04}, $H_{04'}$, $H_{04''}$), 5.35 (4H, s, H_{01}), 4.95 (4H, s, H_7). ^{13}C NMR (75MHz; solvent acetone-d_6) δ 158.8, 156.0, 152.8 (C_6, $C_{6''}$), 148.3, 141.0 (C_{03}, $C_{03'}$, $C_{03''}$), 140.5, 138.5 (C_4, $C_{4''}$),

136.4, 131.2 (C_{05}, $C_{05'}$, $C_{05''}$), 128.9 ($C_{3'''}$, $C_{5'''}$) , 128.0 ($C_{2'''}$, $C_{2'''}$, C_5, $C_{5''}$), 125.0 (C_3, $C_{3''}$), 121.7 ($C_{3'}$, $C_{5'}$). 106.4 (C_{05}, $C_{05'}$, $C_{05''}$), 73.4 (C_{01}), 73.06 (C_7). IR (KBr, cm^{-1}): 108.8, 106.4(C_{04}, $C_{04'}$, $C_{04''}$), 73.4 (C_{01}), 73.0 (C_7). IR (KBr): $\tilde{\nu}/cm^{-1}$ = 2960 w, 2920 w, 2850 w,1609 msh, 1408 m, 1204 w, 849 ssh, 787 msh, 756 msh, 559 ssh. ESI-MS: *m/z* 1377.6 ($[M\text{-}PF_6]^+$), 616.3 ($[M\text{-}2PF_6]^{2+}$). UV-vis (CH_3CN): λ_{max} (ε) = 284.9 (68077), 310.0 (62692), 490.1 (22039) nm (L mol^{-1} cm^{-1}).

[(ttp)Ru(pzt)]$(PF_6)_2$, **4.5**.

[Ru(ttp)Cl_3], **4.3**, (0.265 g, 0.50 mmol) was added to pzt , **1.71**, (0.283 g, 0.50 mmol) in MeOH (20 mL). 10 drops of *N*-methylmorpholine was added. The solution was heated to reflux with stirring for 1 hr. The resulting deep-red solution was filtered through Celite to remove any unchanged [Ru(ttp)Cl_3] and excess methanolic solution of ammonium hexafluorophosphate was added to the filtrate to precipitate the ligand complexes. Further purification was achieved by column chromatography over silica eluting with CH_3CN-saturated KNO_3 solution-H_2O (17: 0.5: 1). The major orange-red band was collected. The complex was isolated as its PF_6^- salt as a red powder. Yield 0.6 g, 94%. ^1H NMR (500MHz; solvent acetone-d_6) δ 9.55 (2H, s, $H_{3'}$(pzt), $H_{5'}$(pzt)), 9.54 (2H, s, $H_{3'}$(ttp), $H_{5'}$(ttp)), 9.18 (4H, d, H_3(pzt, ttp)), $H_{3''}$(pzt, ttp)), 8.41 (2H, d, $H_{2'''}$(pzt), $H_{6'''}$(pzt)), 8.37 (2H, d, $H_{2'''}$, $H_{6'''}$), 8.22 (4H, m, H_4, $H_{4''}$(pzt, ttp)), 7.94 (4H, m, H_6, $H_{6''}$(pzt, ttp)), 7.79 (3H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.74 (2H, d, $H_{3'''}$, $H_{5'''}$(pzt)), 7.70 (2H, d, $H_{3'''}$, $H_{5'''}$(ttp)), 7.69 (3H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.47 (4H, m, H_5, $H_{5''}$(pzt, ttp)), 6.55 (3H, m, H_{04}, $H_{04'}$, $H_{04''}$), 5.34 (2H, s, H_{01}), 4.95 (2H, s, H_7), 2.65 (3H, CH_3 (ttp)). ^{13}C NMR (75MHz; solvent acetone-d_6) δ 158.82, 158.74, 156.03, 155.93, 152.82 (C_6, $C_{6''}$), 148.56, 148.19, 141.03 (C_{03}, $C_{03'}$, $C_{03''}$), 140.43, 138.44 (C_4, $C_{4''}$), 136.37, 133.95, 131.16 (C_{05}, $C_{05'}$, $C_{05''}$), 130.43 ($C_{3'''}$(ttp), $C_{5'''}$(ttp)), 128.92 ($C_{3'''}$(pzt), $C_{5'''}$(pzt)) , 128.00

($C_{2'''}$(pzt), $C_{6'''}$(pzt), C_5(pzt), $C_{5''}$(pzt)), 127.96 ($C_{2'''}$(ttp), $C_{6'''}$(ttp), C_5(ttp), $C_{5''}$(ttp)), 125.00 (C_3, $C_{3''}$), 121.68 ($C_{3'}$(pzt), $C_{5'}$(pzt)), 121.34 ($C_{3'}$(ttp), $C_{5'}$(ttp)), 106.37 (C_{05}, $C_{05'}$, $C_{05''}$), 73.37 (C_{01}), 73.06 (C_7), 20.69 (CH_3(ttp)). IR (KBr): $\tilde{\nu}$/cm^{-1} = 3080 w, 2950 w, 2852 w, 1605 ssh, 1520 m, 1408 msh, 1327 m, 1246 w, 1196 w, 1096 m, 1030 wsh, 949 w, 840 ssh, 787 ssh, 752 msh, 656 wsh, 617 w, 559 ssh, 509 w. ESI-MS: *m/z* 1135.5 ([M-PF_6]$^+$), 495.2 ([M-2PF_6]$^{2+}$). UV-vis (CH_3CN): λ_{max} (ε) = 284.9 (60871), 310.0 (60406), 490.1 (21344) nm (L mol^{-1} cm^{-1}).

[Fe(pzt)$_2$](PF_6)$_2$.2H_2O, **4.6**.

$FeCl_2.4H_2O$ (0.050 g, 0.250 mmol) in ethanol (20 mL) was added to a solution of ligand pzt, **1.71**, (0.283 g, 0.500 mmol) in CH_2Cl_2 : EtOH (1:1) (20 mL) to give a deep purple solution, immediately. The mixture was stirred at room temperature for 1 hr before the CH_2Cl_2 was evaporated *in vacuo*, then to the mixture was added excess of NH_4PF_6 solution in water (5 mL). The purple precipitate was collected by filtration through Celite, dissolved in CH_3CN and was purified by column chromatography (SiO2 eluting with CH_3CN/saturated aqueous KNO_3/water (17:0.5:1). An excess of NH_4PF_6 was added to the major purple fraction and reduced in volume. The precipitate was collected by filtration through Celite, dissolved in CH_3CN and evaporated to dryness to give [Fe(pzt)$_2$](PF_6)$_2$ as a dark purple powder. Further purification was achieved by recrystallisation from CH_3CN-H_2O solution of the complex. Yield 0.32 g, 87%. ^{1}H NMR (500MHz; solvent acetone-d_6) δ 9.74 (4H, s, $H_{3'}$, $H_{5'}$), 9.15 (4H, d, H_3, $H_{3''}$), 8.51 (4H, d, $H_{2'''}$, $H_{6'''}$), 8.18 (4H, m, H_4, $H_{4''}$), 7.80 (6H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.78 (4H, d, $H_{3'''}$, $H_{5'''}$), 7.70 (6H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.68 (4H, d, H_6, $H_{6''}$), 7.37 (4H, m, H_5, $H_{5''}$), 6.55 (6H, t, H_{04}, $H_{04'}$, $H_{04''}$), 5.36 (4H, s, H_{01}), 4.97 (4H, s, H_7). ^{1}H NMR (500MHz; solvent dmso-d_6) δ 9.75 (4H, s, $H_{3'}$, $H_{5'}$), 9.14 (4H, d, H_3, $H_{3''}$), 8.60 (4H, d,

$H_{2'''}$, $H_{6'''}$), 8.12 (4H, m, H_4, $H_{4''}$), 7.83 (6H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.70 (4H, d, $H_{3'''}$, $H_{5'''}$), 7.67 (6H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.35 (4H, d, H_6, $H_{6''}$), 7.28 (4H, m, H_5, $H_{5''}$), 6.57 (6H, t, H_{04}, $H_{04'}$, $H_{04''}$), 5.23 (4H, s, H_{01}), 4.91 (4H, s, H_7). ^{13}C NMR (75MHz; solvent acetone-*d*6) δ 160.90, 158.64, 153.48, 150.41, 141.02, 140.86, 139.16, 136.21, 131.17, 129.00, 128.16, 127.86, 124.33, 121.66, 106.37, 90.03, 73.40, 73.07. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3020 w, 2960 w, 2850 w, 1612 m, 1516 m, 1393 m, 1335 m, 1250 w, 1204 m, 1096 msh, 1018 w, 985 msh, 841 ssh, 791 msh, 752 msh, 559 ssh. ESI-MS: *m/z* 1331.6 ([M-PF_6]$^+$), 593.2 ([M-2PF_6]$^{2+}$). UV-vis (CH_3CN): λ_{max} (ε) = 215.0 (134818), 284.9 (86773), 320.2 (59364), 569.9 (22556) nm (L mol^{-1} cm^{-1}). Anal. Calc. for $C_{66}H_{54}F_{12}FeN_{18}O_2P_2.2H_2O$ (1513.07): C 52.39, H 3.86, N 16.66%; found: C 52.29, H 3.82, N 16.40%.

[Cu(pzt)(NO_3)$_2$], **4.7**.

A solution of Cu(NO_3)$_2$ (0.01 g, 0.04 mmol) in CH_3CN (1 mL) was layered onto a solution of ligand pzt, **1.71**, (0.023 g, 0.04 mmol) in CH_2Cl_2 (1 mL). After about a week green prismatic crystals suitable for X-ray diffraction were obtained upon slow evaporation at 4 ºC. The crystals were separated from the solution by filteration, washed with acetone, ether, then air-dried. Yield 82%. Samples were dried over P_4O_{10} under vacuum for elemental analysis. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3078 w, 1605 msh, 1558 w, 1474 msh, 1381 ssh, 1319 m, 1304 m, 1204 w, 1096 m, 1018 w, 949 w, 918 w, 872 m, 787 m, 656 w, 617 w, 532 w, 409 w. ESI-MS: *m/z* 690.1 ([M-NO_3]$^+$). UV-vis (dmso): λ_{max} (intensity)= 280.0 (99), 290.1 (100), 299.9 (94), 318.1 (65), 349.4 (42), 733.9 (1) nm (%).

[Zn(pzt)Cl_2], **4.8**.

Mehod A: To the ligand pzt, **1.70**, (0.011 g, 0.02 mmol) in CH_2Cl_2 (5 mL) was added $ZnCl_2$ (0.006 g, 0.02 mmol) in CH_3OH (5 mL) dropwise under Ar atmosphere. The reaction mixture was stirred at r.t for 24 hours during which time a white precipitate was formed. The precipitate was collected, washed thoroughly with diethyl ether, then dried under vacuum. Yield 0.009 g, 64%.

Mehod B: To the ligand pzt, **1.70**, (0.011 g, 0.02 mmol) in CH_2Cl_2 (5 mL) was added $ZnCl_2$ (0.006 g, 0.02 mmol) in CH_3CN (5 mL) dropwise under Ar atmosphere. The reaction mixture was stirred at r.t for 24 hours. The clear solution was filtered. Colourless blocks of crystals suitable for X-ray were obtained upon slow evaporation of the solution at r.t. The crystals were separated by filtration, washed with diethyl ether, then dried under vacuum. Yield 0.008 g, 57%. ^{1}H NMR (500MHz; solvent dmso-d_6) δ 9.09(4H, s, $H_{3'}$, $H_{5'}$), 8.97 (4H, d, H_3, $H_{3''}$), 8.95 (4H, d, H_6, $H_{6''}$), 8.39 (4H, dd, H_4, $H_{4''}$), 8.27 (4H, d, $H_{2'''}$, $H_{6'''}$), 7.95 (4H, dd, H_5, $H_{5''}$), 7.81 (6H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.65 (6H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.48 (4H, d, $H_{3'''}$, $H_{5'''}$), 6.54 (6H, m, H_{04}, $H_{04'}$, $H_{04''}$), 5.19 (4H, s, H_{01}), 4.78 (4H, s, H_7).^{13}C NMR (75MHz; solvent dmso-d_6) δ 153.86, 148.77, 147.14, 141.05, 140.80, 140.40, 134.70, 131.16, 128.11, 127.42, 122.80, 120.10, 106.62, 89.39, 72.73, 72.21. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3141 m, 3121 m, 3059 m, 2939 w, 2862 w, 1601 m, 1585 ssh, 1566 w, 1551 m, 1516 m, 1466 m, 1420 m, 1389 ssh, 1323 s, 1276 w, 1250 m, 1204 m, 1126 s, 1096 ssh, 1061 w, 1042 m, 1015 msh, 987 w, 949 msh, 918 m, 868 s, 833 m, 791 ssh, 764 s, 691 w, 660 m, 644 w, 613 m, 521 w. ESI-MS: *m/z* (intensity (%), fragment) 1231.9 (1, $[ZnL_2\text{-}Cl]^+$), 666.2 (2, $[ZnL\text{-}Cl]^+$), 597.3 (100, $[ZnL_2\text{-}2Cl]^{2+}$). Anal. Calc. for $C_{33}H_{27}ClN_9OZn$ (701.92): C 56.47, H 3.88, N 17.96%; found: C 56.56, H 3.79, N 17.77%.

[Cd(pzt)$(NO_3)_2$], **4.9**.

To the ligand pzt, **1.71**, (0.011 g, 0.02 mmol) in CH_2Cl_2 (5 mL) was added $Cd(NO_3)_2.4H_2O$ (0.006 g, 0.02 mmol) in CH_3CN (5 mL) dropwise under an Ar atmosphere. The reaction mixture was stirred at r.t overnight. The clear solution was filtered. Colourless crystals were obtained upon slow evaporation of the solution at r.t. The crystals were separated by filteration, washed with diethyl ether, then dried under vacuum. Yield 0.009 g, 56%. ^{1}H NMR (500MHz; solvent dmso-d_6) δ 9.15 (4H, s, $H_{3'}$, $H_{5'}$), 9.11 (4H, d, H_3, $H_{3''}$), 8.85 (4H, d, H_6, $H_{6''}$), 8.45 (4H, d, H_4, $H_{4''}$), 8.31 (4H, d, $H_{2'''}$, $H_{6'''}$), 7.98 (4H, t, H_5, $H_{5''}$), 7.79 (6H, d, H_{03}, $H_{03'}$, $H_{03''}$), 7.63 (6H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.55 (4H, d, $H_{3'''}$, $H_{5'''}$), 6.53 (6H, t, H_{04}, $H_{04'}$, $H_{04''}$), 5.17 (4H, s, H_{01}), 4.80 (4H, s, H_7).^{13}C NMR (75MHz; solvent dmso-d_6) δ 153.25, 149.76, 149.53, 148.66, 141.22, 141.10, 140.32, 135.29, 131.30, 128.42, 128.27, 127.46, 124.04, 121.06, 106.79, 89.51, 72.82, 72.39. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3113 m, 3078 m, 2947 w, 2893 w, 1701 w, 1601 m, 1574 m, 1551 m, 1516 m, 1481 ssh, 1447 ssh, 1385 ssh, 1296 ssh, 1265 ssh, 1204 m, 1165 m, 111m, 1015 msh, 949 m, 918 m, 895 w, 818 m, 791 ssh, 752 m, 660 w, 640 w, 617 w, 513 w, 451 w. ESI-MS: *m/z* (intensity (%), fragment), 1305.3 (1, [Cd$(pzt)_2$-NO_3]$^+$), 622.3 (100, [Cd$(pzt)_2$-$2NO_3$]$^{2+}$). Anal. Calc. for $C_{33}H_{27}CdN_{11}O_7$ (802.05): C 49.42, H 3.39, N 19.21%; found: C 49.43, H 3.40, N 18.83%.

{{[Fe$(pzt)_2$]$(NO_3)_2$}$(AgNO_3)_2$}$_\infty$, **4.10**.

To complex **4.6** (0.001 g, 0.0007 mmol) in acetone (1 mL) was added $AgNO_3$ (0.0003 g, 0.0014 mmol) in acetonitrile (1 mL), dropwise. The mixture was allowed to slowly evaporate in the air at r.t. The microcrystalline compound which was crystallized from the reaction mixture was collected, washed with diethyl ether, and air-dried. ^{1}H NMR (500MHz; solvent acetone-d_6) δ 9.67 (4H, s, $H_{3'}$, $H_{5'}$), 9.10 (4H, d,

H_3, $H_{3''}$), 8.53 (4H, d, $H_{2'''}$, $H_{6'''}$), 8.21 (6H, d, H_{03}, $H_{03'}$, $H_{03''}$), 8.15 (4H, m, H_4, $H_{4''}$), 8.06 (6H, d, H_{05}, $H_{05'}$, $H_{05''}$), 7.78 (4H, d, $H_{3'''}$, $H_{5'''}$), 7.66 (4H, d, H_6, $H_{6''}$), 7.34 (4H, m, H_5, $H_{5''}$), 6.8 (6H, t, H_{04}, $H_{04'}$, $H_{04''}$), 5.49 (4H, s, H_{01}), 5.04 (4H, s, H_7). ESI-MS: *m/z* 763.26 ($\{[Fe(pzt)_2Ag_2)](NO_3)_2\}^{2+}$).

$\{\{[Fe(pzt)_2](ClO_4)_2\}(AgClO_4)_2\}_\infty$, **4.11**.

The same method was used as above except $AgClO_4$ in acetone was applied instead. ESI-MS: *m/z* 801.23 ($\{[Fe(pzt)_2Ag_2)](ClO_4)_2\}^{2+}$).

$\{\{[Fe(pzt)_2](BF_4)_2\}(AgBF_4)\}_\infty$, **4.12**.

To complex **4.6** (0.001 g, 0.0007 mmol) in acetone (1 mL) was added $AgBF_4$ (0.0003 g, 0.0014 mmol) in acetone (1 mL), dropwise. The mixture was allowed to slowly evaporate in the air at r.t. The microcrystalline compound which was crystallized from the reaction mixture was collected, washed with small amount of cold acetone, and air-dried. ESI-MS: *m/z* 691.4 ($\{[Fe(pzt)_2Ag)](BF_4)\}^{2+}$).

$[(ttp)Ru(bptt)](NO_3)_2$, **5.36**.

$[Ru(ttp)Cl_3]$, **4.3**, (0.187 g, 0.353 mmol) was added to the ligand bptt, **5.32**, (0.242 g, 0.353 mmol) in dry MeOH (20 mL) under Ar atmosphere. 10 drops of *N*-methylmorpholine was added. The solution was heated to reflux with stirring for 2 hr, then was cooled to r.t. The resulting deep-red solution was filtered through Celite to remove any unchanged $[Ru(ttp)Cl_3]$ and excess methanolic solution of ammonium hexafluorophosphate was added to the filtrate to precipitate the ligand complexes. Further purification was achieved by column chromatography over silica eluting with CH_3CN-saturated KNO_3 solution-H_2O (7:0.5:1). The major orange-red band was

collected. The NO_3^- salt of this complex was used in the subsequent reaction. ^{1}H NMR (500MHz; solvent dmso-d_6) δ 9.58 (2H, s, $H_{3'}$(bptt), $H_{5'}$(bptt)), 9.49 (2H, s, $H_{3'}$(ttp), $H_{5'}$(ttp)), 9.23 (4H, d, H_3(bptt, ttp)), $H_{3''}$(bptt, ttp)), 8.49 (2H, d, $H_{2'''}$(ttp), $H_{6'''}$(ttp)), 8.26 (2H, d, $H_{2'''}$(bptt), $H_{6'''}$(bptt), 8.20-8.15 (4H, m, H_4(bptt, ttp), $H_{4''}$(bptt, ttp)), 7.94-7.92 (4H, m, aromatic, phthalimide), 7.85-7.83 (4H, m, aromatic, phthalimide), 7.69-7.65 (6H, m, H_6(bptt, ttp), $H_{6''}$(bptt, ttp), $H_{3'''}$(ttp), $H_{5'''}$(ttp)), 7.58 (2H, d, $H_{3'''}$(bptt), $H_{5'''}$(bptt)), 7.41-7.38 (4H, m, H_5(bptt, ttp), $H_{5''}$(bptt, ttp)), 3.92 (2H, s, H_7), 3.82 (4H, m, H_{02}, $H_{02'}$), 2.90 (4H, m, H_{01}, $H_{01'}$), 2.62 (3H, s, CH_3). ^{13}C NMR (75MHz; solvent dmso-d_6) δ 206.71, 167.96, 158.21, 155.17, 152.33, 146.99, 146.60, 141.67, 140.46, 138.17, 134.38, 133.26, 131.85, 130.14, 129.56, 127.86, 127.67, 127.27, 124.97, 122.98, 120.82, 56.96, 51.59, 35.50, 21.11. ESI-MS: *m/z* (intensity (%), fragment) 1171.5535 (1, $[M-(NO_3)]^+$), 554.8108 (58, $[M-2(NO_3)]^{2+}$), 370.2076 (100, $[M+H-2(NO_3)]^{3+}$). UV-vis (DMSO): λ_{max} (ε) = 229.9 (37263), 240.0 (40974), 250 (41554), 265.1 (42072), 500.0 (3761) nm (L mol^{-1} cm^{-1}). To obtain the yield of the reaction, complex was isolated as its PF_6^- salt as a red powder. Yield 0.39 g, 80%. The same sample was submitted for elemental analysis. Anal. Calc. for $C_{64}H_{49}F_{12}N_9O_4P_2Ru.3H_2O$ (1453.18): C 52.90, H 3.81, N 8.67%; found: C 53.14, H 3.52, N 8.62%.

[(ttp)Ru(dint)+H](PF$_6$)$_3$, **5.37**.

Complex **5.36** (containing bptt) (0.12 g, 0.1 mmol) in MeOH (10 mL) was added into a MeOH solution of NH_2NH_2 (0.5 M, 10 mL), and the mixture was stirred overnight at room temperature, filtered, and to the filtrate was added excess aqueous NH_4PF_6 solution, then the volume of the mixture was reduced under vacuum to about 10 mL. The orange-red precipitate was collected by filtration through Celite, washed

with water, diethyl ether, and recrystallised from acetonitrile-water(1:2) to afford a red powder. Yield 0.1 g, 91%. ^{1}H NMR (500MHz; solvent dmso-d_6) δ 9.57 (2H, s, $H_{3'}$(ttp), $H_{5'}$(ttp)), 9.56 (2H, s, $H_{3'}$(dint), $H_{5'}$(dint)), 9.21 (4H, d, H_3(dint, ttp)), $H_{3''}$(dint, ttp)), 8.50-8.47 (4H, m, $H_{2'''}$(ttp, dint), $H_{6'''}$(ttp, dint)), 8.18-8.15 (4H, m, H_4(dint, ttp), $H_{4''}$(dint, ttp)), 7.83 (2H, d, $H_{3'''}$(ttp), $H_{5'''}$(ttp)), 7.69 (2H, d, $H_{3'''}$(dint), $H_{5'''}$(dint)), 7.66-7.63 (4H, m, H_6(dint, ttp), $H_{6''}$(dint, ttp), 7.39-7.36 (4H, m, H_5(dint, ttp), $H_{5''}$(dint, ttp)), 3.92 (2H, s, H_7), 3.85 (4H, m, H_{02}, $H_{02'}$), 2.71 (4H, m, H_{01}, $H_{01'}$), 2.62 (3H, s, CH_3). IR (KBr): $\tilde{\nu}$/cm^{-1} = 3660 m, 3286 w, 3082 w, 2908 w, 2846 w, 1693 m, 1609 m, 1531 w, 1477 m, 1427 w, 1408 m, 1362 w, 1288 w, 1246 w, 1196 w, 1165 w, 1130 w, 1088 w, 1030 w, 972 w, 845 ssh, 787 s, 752 m, 702 w, 656 w, 621 w, 559 s, 513 m, 486 w, 424 w, 405 m. ESI-MS: *m/z* (intensity (%), fragment) 497.6301 (11, $[M+H-(PF_6)]^{2+}$), 424.6406, (22, $[M-2(PF_6)]^{2+}$), 283.4246 (100, $[M+H-2(PF_6)]^{3+}$). UV-vis (DMSO): λ_{max} (ε) = 215.1 (32358), 224.9 (30968), 245.0 (33576), 255 (32904), 500.0 (5319) nm (L mol^{-1} cm^{-1}). Anal. Calc. for $C_{48}H_{46}F_{18}N_9P_3Ru$ (1284.90): C 44.87, H 3.61, N 9.81%; found: C 44.78, H 3.55, N 9.56%.

[(ttp)Ru(bpat)]$(PF_6)_2$, **5.38**.

Ligand bpat, **531**, (0.104 g, 0.20 mmol) and [Ru(ttp)Cl_3], **4.3**, (0.106 g, 0.20 mmol) in dry MeOH (50 mL) in presence of *N*-methylmorpholine (12 drops) were heated at refulx for 1h under Ar. The deep red solution was cooled to room temperature before it was filtered to remove any unreacted materials and excess methanolic solution of ammonium hexafluorophosphate was added to the filtrate to precipitate the ligand complexes. The dark red precipitate was dissolved in CH_3CN, then the solution was taken to dryness under vacuum and the residue was purified by column chromatography (SiO2 eluting with CH_3CN-saturated KNO_3 solution-H_2O, 14

:0.5 :1). An excess of NH_4PF_6 was added to the major red fraction and the solution was reduced in volume under vacuum. The precipitate was collected by filtration over Celite, before it was dissolved in CH_3CN, and evaporated to dryness *in vacuo* to afford [(ttp)Ru(bpat)]$(PF_6)_2$ as a red powder. Yield 0.042g, 17 %. ^{1}H NMR (500MHz; solvent acetone-d_6) δ 9.53 (2H, s, $H_{3'}$(ttp), $H_{5'}$(ttp)), 9.49 (2H, s, $H_{3'}$(bpat), $H_{5'}$(bpat)), 9.17-914 (4H, m, H_3(bpat, ttp)), $H_{3''}$(bpat, ttp)), 8.95 (2H, d, H_{04}(bpat), $H_{04'}$(bpat)), 8.43 (2H, d, $H_{2'''}$(bpat), $H_{6'''}$(bpat)), 8.37 (2H, d, $H_{2'''}$(ttp), $H_{6'''}$(ttp)), 8.22-8.19 (4H, m, H_4(bpat, ttp), $H_{4''}$(bpat, ttp)), 8.16 (2H, m, H_{06}(bpat), $H_{06'}$(bpat)), 7.99 (2H, d, $H_{3'''}$(bpat), $H_{5'''}$(bpat)), 7.93 (2H, d, H_6(bpat or ttp), $H_{6''}$(bpat or ttp)), 7.90 (2H, d, H_6(bpat or ttp), $H_{6''}$(bpat or ttp)), 7.84 (2H, d H_{07}(bpat), $H_{07'}$(bpat)), 7.70 (2H, d, $H_{3'''}$(ttp), $H_{5'''}$(ttp)), 7.66 (2H, m, H_{05}(bpat), $H_{05'}$(bpat)), 7.47-7.43 (4H, m, H_5(bpat, ttp), $H_{5''}$(bpat, ttp)), 4.53 (4H, s, H_{01}, $H_{01'}$), 4.45 (2H, s, H_7), 2.65 (3H, s, CH_3). ^{13}C NMR (75MHz; solvent CD_3CN) δ 159.22, 159.16, 156.91, 156.48, 156.33, 153.41, 149.34, 148.55, 148.01, 141.97, 140.67, 139.47, 138.98, 137.57, 134.90, 131.66, 131.26, 128.93, 128.66, 128.47, 128.41, 125.54, 125.49, 125.03, 124.80, 122.46, 122.33, 59.38, 58.61, 21.43. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3647 w, 3020 w, 1607 m, 1574 w, 1468 m, 1431 m, 1406 m, 1288 w, 1248 w, 1086 m, 1030 w, 847 ssh, 791 m, 756 w, 667 w, 656 w, 619 w, 557 s, 529 w, 513 w, 492 m, 480 w, 465 w, 446 m, 422 m, 418 w. ESI-MS: *m/z* (intensity (%), fragment) 545.6792 (5, $[M+H-(PF_6)]^{2+}$), 472.7060, (19, $[M-2(PF_6)]^{2+}$), 315.4781 (100, $[M+H-2(PF_6)]^{3+}$). UV-vis (CH_3CN): λ_{max} = 285.0, 310.0, 490.0 nm.

[(ttp)Ru(H+**5.19**)]$(PF_6)_3$, **5.39**.

To ligand **5.18** (0.347 g, 0.641 mmol) in MeOH (30 mL) was added [Ru(ttp)Cl_3], **4.3**, (0.340 g, 0.641 mmol) and *N*-methylmorpholine (10 drops). The

mixture was heated at reflux overnight under Ar. The deep red solution was cooled to room temperature before it was filtered. Excess methanolic solution of ammonium hexafluorophosphate was added to the filtrate to precipitate the complexes. The dark red precipitate was dissolved in CH_3CN, then the solution was taken to dryness under vacuum and the residue was purified by column chromatography (SiO2 eluting with CH_3CN-saturated KNO_3 solution-H_2O, 7 :0.5 :1). The major red fraction was collected and the solution was taken to dryness under vacuum. Further purification was achieved by recrystallisation from MeOH-nitromethane (1:2) to afford [(ttp)Ru(**5.19**)]$(NO_3)_2$ as a red-orange microcrystals. The product was isolated as its PF_6^- salt as a red-orange microcrystalline material. Yield 0.45 g, 53%. ^{1}H NMR (300MHz; solvent acetone-d_6) δ 9.53 (4H, s), 9.17-914 (4H, m), 8.50-8.36 (4H, m), 8.26-8.00 (5H, m), 7.97-7.92 (8H, m), 7.70 (2H, d), 7.47-7.43 (4H, m), 4.26-4.18(2H, (AB), PhCH_2N), 3.18-412 (12H, macrocycle), 2.65 (3H, CH_3). IR (KBr): $\tilde{\nu}$/cm^{-1} = 3653 m, 1670 m, 1607 m, 1578 w, 1477 m, 1466 m, 1431 m, 1406 m, 1385 s, 1290 w, 1246 w, 1194 w, 1163 w, 1084 w, 1055 w, 1029 w, 970 w, 847 ssh, 789 s, 756 m, 733 w, 669 w, 656 w, 617 w, 557 ssh, 509 w, 500 w, 480 w, 446 w, 422 m. ESI-MS: *m/z* (intensity (%), fragment) 1048.3851 (0.5, $[M-(PF_6)]^+$), 524.7050 (29, $[M+H-(PF_6)]^{2+}$), 451.6993, (29, $[M-2(PF_6)]^{2+}$), 301.4705 (100, $[M+H-2(PF_6)]^{3+}$). UV-vis (CH_3CN): λ_{max} (ε) = 285.0 (32115), 305.0 (34418), 490.0 (12744) nm (L mol^{-1} cm^{-1}). Anal. Calc. for $C_{51}H_{48}F_{18}N_9OP_3Ru$ (1338.95): C 45.75, H 3.61, N 9.41%; found: C 45.34, H 3.80, N 9.50%.

[(ttp)Ru(tcnt)]$(NO_3)_2$, **5.40**.

Complex precursor [(ttp)Ru(H+**5.19**)]$(PF_6)_3$, **5.39**, (0.4 g, 0.3 mmol) in acetone (10 mL) was subjected to base hydrolysis by refluxing the solution in presence of

KOH (1 g, 18 mmol) in water (10 mL) for 4 days. The reaction progress was monitored using TLC and ^{1}H NMR. The reaction mixture was cooled at r.t, before the acetone was taken off the solution by rotary evaporation. The precipitate was collected by filtration over Celite and the residue was purified by column chromatography (SiO2 eluting with CH_3CN-saturated KNO_3 solution-H_2O, 7:0.5:1). The major red fraction was collected, and taken to dryness by rotary evaporation, then the complex was dissolved in small amount of MeOH, then filtered through Celite to remove KNO_3. This was repeated three times. The solution was taken to dryness on vacuum, then was recrystallised by slow evaporation of the MeOH-benzene (1:2) solution of the complex.. The precipitate was filtered, washed well with benzene, diethyl ether, and air-dried to afford a red-orange powder. Yield 0.2 g, 50%. The unreacted starting materials were also collected and reused. ^{1}H NMR (500MHz; solvent dmso-d_6) δ 9.59 (2H, s, $H_{3'}$(tcnt), $H_{5'}$(tcnt)), 9.57 (2H, s, $H_{3'}$(ttp), $H_{5'}$(ttp)), 9.23-9.21 (4H, m, H_3(tcnt, ttp)), $H_{3''}$(tcnt, ttp)), 8.55 (2H, d, $H_{2'''}$(tcnt), $H_{6'''}$(tcnt)), 8.48 (2H, d, $H_{2'''}$(ttp), $H_{6'''}$(ttp)), 8.17-8.16 (4H, m, H_4(tcnt, ttp), $H_{4''}$(tcnt, ttp)), 7.88 (2H, d, $H_{3'''}$(tcnt), $H_{5'''}$(tcnt)), 7.69 (2H, d, $H_{3'''}$(ttp), $H_{5'''}$(ttp)), 7.66-7.64 (4H, m, H_6(tcnt, ttp), $H_{6''}$(tcnt, ttp), 7.40-7.37 (4H, m, H_5(tcnt, ttp), $H_{5''}$(tcnt, ttp)), 4.08 (2H, s, H_7), 3.56 (4H, br, H_{10}, $H_{10'}$), 3.27 (4H, br, H_9, $H_{9'}$), 3.03 (4H, br, H_8, $H_{8'}$), 2.62 (3H, s, CH_3). ^{13}C NMR (75MHz; solvent dmso-d_6) δ 158.21, 158.14, 155.24, 155.13, 152.31 (C_6, $C_{6''}$ (tcnt, ttp)), 147.04, 146.65, 140.48, 138.18 (C_4, $C_{4''}$ (tcnt, ttp)), 135.38, 133.26, 130.91 ($C_{3'''}$, $C_{5'''}$ (tcnt)),130.15 ($C_{3'''}$, $C_{5'''}$ (ttp)), 127.84 ($C_{2'''}$, $C_{6'''}$(tcnt)), 127.68 ($C_{3'''}$, $C_{5'''}$(ttp)), 124.97 (C_3, $C_{3''}$(tcnt, ttp)), 121.17 ($C_{3'}$, $C_{5'}$(tcnt)), 120.86 ($C_{3'}$, $C_{5'}$(ttp)), 58.47 (C_7), 48.82 (C_8, $C_{8'}$), 44.19 (C_9, $C_{9'}$), 43.16 (C_{10}, $C_{10'}$), 21.12 (CH_3). The product was also isolated as its PF_6^- salt. Samples were submitted for further characterisations. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3655 m, 3000 mbr, 1607 m, 1533 w, 1522 w, 1479 m, 1466 m, 1431 m, 1406 m, 1358 w, 1288 w, 1246 w, 1194 w,

1163 w, 1121 w, 1084 w, 1055 w, 1030 m, 968 w, 847 ssh, 789 ssh, 754 m, 741 w, 721 w, 656 w, 619 w, 557 ssh, 509 w, 480 w, 444 w, 434 w, 422 m. ESI-MS: *m/z* (intensity (%), fragment) 510.6956, (23, $[M\text{-}2(PF_6)]^{2+}$), 292.1342 (100, $[M\text{-}3(PF_6)]^{3+}$). UV-vis (CH_3CN): λ_{max} = 285.0, 310.0, 490.0 nm. Anal. calc. for $C_{50}H_{49}F_{24}N_9P_4Ru.3H_2O$ (1510.96): C 39.75, H 3.67, N 8.34%; found: C 39.22, H 3.63, N 8.69%.

[(ttp)Ru(ptmtb)]$(PF_6)_3$, **5.41**.

To ligand ptmtb, **5.34**, (0.026 g, 0.04 mmol) in MeOH (15 mL) was added [Ru(ttp)Cl_3], **3.2**, (0.022 g, 0.04 mmol) and *N*-methylmorpholine (5 drops). The mixture was heated at reflux overnight under Ar. The deep red solution was cooled to room temperature before it was filtered. Excess methanolic solution of ammonium hexafluorophosphate was added to the filtrate to precipitate the complexes. The dark red precipitate was dissolved in CH_3CN, then the solution was taken to dryness under vacuum and the residue was purified by column chromatography (SiO2 eluting with CH_3CN-saturated KNO_3 solution-H_2O, 10 :1 :2). The major red fraction was collected and the solution was taken to dryness under vacuum. to afford [(ttp)Ru(ptmtb)]$(NO_3)_3$ as a red-orange microcrystals. The product was isolated as its PF_6^- salt as a red-orange microcrystalline material. Yield 0.043 g, 78%. ^{13}C NMR (75 MHz; solvent CD_3CN) δ 159.21, 159.10, 156.69, 156.28, 153.42, 149.43, 147.56, 141.99, 140.10, 139.02, 135.40, 134.88, 134.82, 131.27, 129.66, 129.48, 129.30, 128.67, 128.54, 128.42, 125.63, 125.53, 122.68, 122.35, 84.35, 71.13, 62.23, 61.10, 55.43, 55.06, 53.28, 53.21, 49.87, 47.69, 43.46, 21.43, 19.83, 19.67. ESI-MS: *m/z* (intensity (%), fragment) 557.2206 (29, $[M\text{-}2(PF_6)]^{2+}$), 323.1544 (100, $[M\text{-}3(PF_6)]^{3+}$). IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3651 w, 2949 w, 2843 w, 1655 w, 1607 m, 1524 w, 1477 m, 1466 m, 1431 m, 1406 m, 1384

m, 1356 w, 1246 w, 1196 w, 1144 w, 1086 w, 1030 w, 845 ssh, 789 s, 754 m, 735 w, 656 w, 557 ssh, 492 w, 480 w, 448 w, 421 w. UV-vis (CH_3CN): λ_{max} = 285.0, 295.0, 305.0, 490.0 nm.

[(ttp)Ru(cymt)]$(PF_6)_2$, **5.30**.

An established literature method[197] was adopted for the preparation of **5.30**. [Ru(ttp)Cl_3], **4.3**, (0.140 g, 0.264 mmol) was added to the ligand cymt, **1.74**, (0.138 g, 0.264 mmol) in dry MeOH (20 mL) under an Ar atmosphere. 10 drops of *N*-methylmorpholine was added. The solution was heated to reflux with stirring for 1 hr then was cooled to r.t. The resulting deep-red solution was filtered through Celite to remove any unchanged [Ru(ttp)Cl_3] and excess methanolic solution of ammonium hexafluorophosphate was added to the filtrate. The volume of the mixture was reduced to *ca*. 10 mL, before it was kept in the refrigerator. The precipitate was collected by filtration, washed with water, diethyl ether, and air-dried. To the CH_3CN solution of the product was added NH_4OH (10 drops) before it was purified by column chromatography over silica eluting with CH_3CN-saturated KNO_3 solution-H_2O (17: 1: 2). The major orange-red band was collected as its PF_6^- salt as a red-orange powder. Yiled 0.082-0.100 g, 25-31% ^{1}H NMR (500MHz; solvent acetone-d_6) δ 9.513 (2H, s, $H_{3'}$(ttp), $H_{5'}$(ttp)), 9.508 (2H, s, $H_{3'}$(cymt), $H_{5'}$(cymt)), 9.16-9.12 (4H, m, H_3(cymt, ttp), $H_{3''}$(cymt, ttp)), 8.57 (2H, d, $H_{2'''}$(ttp), $H_{6'''}$(ttp)), 8.39 (2H, d, $H_{2'''}$(cymt), $H_{6'''}$(cymt)), 8.21-8.16 (4H, m, H_4(cymt, ttp), $H_{4''}$(cymt, ttp)), 7.93-7.89 (4H, m, H_6(cymt, ttp), $H_{6''}$(cymt, ttp)), 7.85 (2H, d, $H_{3'''}$(ttp), $H_{5'''}$(ttp)), 7.69 (2H, d, $H_{3'''}$(cymt), $H_{5'''}$(cymt)), 7.47-7.43 (4H, m, H_5(cymt, ttp), $H_{5''}$(cymt, ttp)), 4.35 (2H, s, H_7), 3.71-314 (20H, m, cyclam), 2.64 (3H, s, CH_3). ^{13}C NMR (75MHz; solvent dmso-d_6) δ 158.83, 158.78, 156.09, 155.97, 152.89, 152.83, 148.57, 147.86, 141.12, 138.43, 136.74, 136.57,

133.98, 131.80, 130.45, 128.38, 128.00, 127.83, 125.06, 124.99, 121.64, 121.35, 54.60, 52.45, 51.63, 50.48, 49.85, 48.30, 47.53, 45.50, 45.27, 24,89, 22.36, 20.71. IR (KBr): $\tilde{\nu}$/cm^{-1} = 3709 w, 3720 w, 2926 m, 2858 m, 1695 w, 1653 w, 1605 m, 1539 w, 1520 w, 1468 m, 1431 m, 1408 m, 1366 w, 1288 w, 1086 w, 1030 w, 845 ssh, 789 s, 756 m, 741 w, 721 w, 656 w, 623 w, 592 w, 557 ssh, 538 w, 519 w, 480 w, 447 w, 421 m, 407 w. ESI-MS: *m/z* (intensity (%), fragment) 546.2617 (22, $[M+H-(PF_6)]^{2+}$), 473.2755 (6, $[M-2(PF_6)]^{2+}$), 315.8028 (100, $[M+H-2(PF_6)]^{3+}$). UV-vis (CH_3CN): λ_{max} = 244.0, 286.0, 310.0, 490.0 nm.

[(ttp)Ru(cynt)]$(PF_6)_2$, **5.43**.

An established literature method[197] was adopted for the preparation of **5.43**. [Ru(ttp)Cl_3], **3.2**, (0.140 g, 0.264 mmol) was added to the ligand cynt, **1.75**, (0.130 g, 0.264 mmol) in dry MeOH (20 mL) under an Ar atmosphere. 10 drops of *N*-methylmorpholine was added. The solution was heated to reflux with stirring for 1 hr then was cooled to r.t. The resulting deep-red solution was filtered through Celite to remove any unchanged [Ru(ttp)Cl_3] and excess methanolic solution of ammonium hexafluorophosphate was added to the filtrate. The volume of the mixture was reduced to *ca*. 10 mL, before it was kept in the refrigerator. The precipitate was collected by filtration, washed with water, diethyl ether, and air-dried. To the CH_3CN solution of the product was added NH_4OH (10 drops) before it was purified by column chromatography over silica eluting with CH_3CN-saturated KNO_3 solution-H_2O (7: 1: 1). The major orange-red band was collected as its PF_6^- salt as a red-orange powder. Yiled 0.084 g, 26% ^{1}H NMR (500MHz; solvent acetone-d_6) δ 9.58 (2H, s), 9.57 (2H, s), 9.25-9.21 (4H, m), 8.55-8.47 (4H, m)), 8.20-8.15 (4H, m), 7.86 (2H, d), 7.70-7.66 (6H, m), 7.43-7.39 (4H, m), 4.07 (2H, s, H_7), 3.37-2.99 (19H, cyclen), 2.60 (3H, s,

CH_3). ^{13}C NMR (75MHz; solvent dmso-d_6) δ 158.27, 158.22, 155.90, 155.27, 155.18, 152.37, 149.56, 147.17, 146.94, 140.48, 138.27, 137.81, 135.71, 133.39, 131.20, 130.23, 128.05, 127.79, 127.19, 125.08, 124.84, 121.43, 120.99, 55.47, 47.57, 45.08, 42.53, 42.32, 21.18. ESI-MS: *m/z* (intensity (%), fragment) 532.4 (12, $[M+H-4(PF_6)]^{2+}$), 459.4 (7, $[M-2(PF_6)]^{2+}$), 306.6 (100, $[M+H-2(PF_6)]^{3+}$). UV-vis (CH_3CN): λ_{max} = 285.0, 305.0, 490.0 nm.

[(ttp)Ru(cymt)Co(NO_2)$_2$](PF_6)$_3$, **5.44**

All opperations were caried out in dark: To complex **5.30** (0.1 g, 0.08 mmol) in acetone (5 mL) was added $Na_3[Co(NO_2)_6]$ (0.032 g, 0.08 mmol) in water (2 mL). Et_3N (10 drops) was added The mixture was gently heated at 50 ºC for 4-6 hr. To the reaction mixture was added excess NH_4PF_6 after it was cooled to r.t. The precipitate was collected by filtration through Celite and washed with water, diethyl ether, and air-dried to afford a red powder. Further purification was achieved by column chromatography over silica eluting with CH_3CN-saturated KNO_3 solution-H_2O (10: 1: 2). The major orange-red band was collected as its PF_6^- salt as a red-orange powder. Yiled 0.103 g, 84%. ^1H NMR (500MHz; solvent acetone-d_6) δ 9.52 (2H, s, $H_{3'}$(ttp), $H_{5'}$(ttp)), 9.50 (2H, s, $H_{3'}$(cymt), $H_{5'}$(cymt)), 9.16-9.13 (4H, m, H_3(cymt, ttp), $H_{3''}$(cymt, ttp)), 8.56 (2H, d, $H_{2'''}$(ttp), $H_{6'''}$(ttp)), 8.41 (2H, d, $H_{2'''}$(cymt), $H_{6'''}$(cymt)), 8.20-8.17 (4H, m, H_4(cymt, ttp), $H_{4''}$(cymt, ttp)), 7.94 (2H, d, $H_{3'''}$(ttp), $H_{5'''}$(ttp)), 7.91 (4H, m, H_6(cymt, ttp), $H_{6''}$(cymt, ttp)), 7.69 (2H, d, $H_{3'''}$(cymt), $H_{5'''}$(cymt)), 7.48-7.43 (4H, m, H_5(cymt, ttp), $H_{5''}$(cymt, ttp)), 4.38 (2H, s, H_7), 4.18-2.24 (20H, m, cyclam), 2.64 (3H, s, CH_3). ^{13}C NMR (75MHz; solvent acetone-d_6) δ 158.67, 158.60, 156.01, 155.76, 152.69 (2C, $C_{3'''}$(ttp), $C_{5'''}$(ttp)), 148.53, 147.40, 140.87, 138.31 (4C, C_4(cymt, ttp), $C_{4''}$(cymt, ttp)), 138.07, 134.00 (2C, C_6(cymt or ttp), $C_{6''}$(cymt or ttp)), 133.91 (2C,

C_6(cymt or ttp), $C_{6''}$(cymt or ttp)), 132.05, 130.33 (2C, $C_{3'''}$(cymt), $C_{5'''}$(cymt)), 128.39 (2C, $C_{2'''}$(ttp), $C_{6'''}$(ttp)), 127.95 (2C, C_5(cymt or ttp), $C_{5''}$(cymt or ttp)), 127.88 (2C, C_5(cymt or ttp), $C_{5''}$(cymt or ttp)), 127.81 (2C, d, $C_{2'''}$(cymt), $C_{6'''}$(cymt)), 124.92 (4C, m, C_3(cymt, ttp), $C_{3''}$(cymt, ttp)), 121.80 (2C, $C_{3'}$(ttp), $C_{5'}$(ttp)), 121.24 (2C, $C_{3'}$(cymt), $C_{5'}$(cymt)), 56.27, 53.75, 52.77, 52.24, 51.87, 50.66, 49.58, 48.37, 46.58, 27.20, 24.75, 20.65 (CH_3). IR (KBr): $\tilde{\nu}$/cm^{-1} = 2924 m, 2855 w, 1607 w, 1558 w, 1466 w, 1406 m, 1385 m, 1271 ssh, 1088 w, 1028 w, 847 ssh, 789 m, 758 w, 648 w, 594 w, 559 m, 515 w, 471 w, 446 w, 434 w, 407 w. ESI-MS: *m/z* (intensity (%), fragment) 621.2722, (48, $[M\text{-}2(PF_6)]^{2+}$), 365.8482 (100, $[M\text{-}3(PF_6)]^{3+}$). UV-vis (CH_3CN): λ_{max} = 285.0, 310.0, 490.0 nm. Anal. Calc. for $C_{54}H_{53}CoF_{18}N_{12}O_4P_3Ru.3H_2O.O(C_2H_3)_2$ (1657.14): C 42.04, H 4.20, N 10.14%; found: C 41.91, H 3.97, N 9.84.

[(ttp)Ru(cymt)Co(Cl)$_2$]Cl$_3$, **5.46**.

All operations were caried out in dark: To an acetone solution of complex **5.44** (0.10 g, 0.065 mmol) was added 6 M HCl (5 mL). The reaction mixture was heated on the steam bath until a precipitate was formed. The red microcrystalline material was collected and washed well with acetone and air-dried. Recrystallisation was acheived by vapour diffusion of diethyl ether into MeOH solution of the complex. A red precipitate which was formed, was collected by filtration though Celite, washed well with diethyl ether, redissolved in MeOH. The solvent was removed on the rotary evaporator to give a red powder. Yield 0.041 g, 53%. ^{13}C NMR (75MHz; solvent methanol-d_4) δ 160.00, 159.94, 157.27, 157.01, 153.60, 153.52, 150.53, 149.32, 142.35, 139.68, 138.92, 135.40, 135.29, 131.62, 129.53, 129.24, 129.15, 126.49, 126.34, 123.16, 122.75, 57.72, 57.44, 54.77, 54.76, 53.85, 52.86, 50.74, 29.11, 26.57, 21.70. ESI-MS: *m/z* (intensity (%), fragment) 556.2232, (17, $[M\text{-}2Cl]^{2+}$), 359.1535

(100, $[M-3Cl]^{3+}$). IR (KBr): $\tilde{\nu}/cm^{-1}$ = 3398 br, 3051 br, 2939 m, 2893 m, 1720 m, 1608 s, 1535 w, 1477 s, 1461 s, 1427 s, 1412 ssh, 1285 w, 1246 m, 1200 m, 1165 w, 1130 m, 1087 m, 1030 s, 984 w, 953 w, 910 w, 883 w, 814 m, 791 ssh, 756 m, 732 w, 702 w, 656 w, 621 w, 509 m, 444 m, 424 w. UV-vis (MeOH): λ_{max} = 285.0, 310.0, 490.0 nm.

$[Ru(\mathbf{2.1})_2](PF_6)_2$, **6.2**.

The ruthenium complex **6.2** was synthesised according to the methods of Hanan and Polson.[209,260] Red block crystals were obtained by slow evaporation of a CH_3CN-H_2O solution of the complex (yield 16%). Found C 42.70, H 2.63, N 16.00%. Calc. for $C_{36}H_{24}F_{12}N_{12}P_2Ru$: C 42.57, H 2.38, N 16.55%.

$[Fe(tpt)_2]Cl_2.4H_2O$, **6.3** and $[Fe(tpt)(Cl)_2]_2(\mu\text{-}O).2H_2O$, **6.4**.

To a solution of tpt in MeOH was added $FeCl_2.4H_2O$ in MeOH dropwise with stirring. A deep purple solution which was formed immediately, was stirred for 4 hr at r.t. The reaction mixture was taken into dryness on vacuum, then was redissolved in water. Dark navy-blue prisms along with some orange rod-like crystals were obtained by slow evaporation of the reaction mixture, successively. The X-ray quality crystals of **6.3** were collected by filtration, washed with diethyl ether and air-dried. ^1H NMR (300MHz; solvent D_2O, TPMS (internal reference)) δ 9.40 (2H, d), 9.23 (4H, d), 9.09 (2H, d), 8.43 (2H, dd), 8.15 (4H, dd), 7.99 (2H, dd), 7.57 (4H, dd), 7.37 (4H, dd). ^{13}C NMR (75MHz; solvent D_2O) δ 178.48, 173.19, 157.55, 156.46, 153.42, 153.16, 142.67, 142.24, 133.07, 131.73, 131.09, 129.44. ESI-MS: *m/z* (intensity (%), fragment) 715.0875 (3, $[M-2(PF_6)]^+$), 340.0758 (100, $[M-2(Cl)]^{2+}$). Found C 52.12, H 4.03, N 20.20%. Calc. for $C_{36}H_{32}Cl_2FeN_{12}O_4$: C 52.51, H 3.92, N 20.41%. Orange rod-

like crystals suitable for X-ray were also obtained from the same reaction mixture. Single crystals were used for X-ray crystallography. Further characterisation of this complex was not pursued due to poor solublity in common solvents.

$[Cu(H_2O)_6](NO_3)_2$, **6.5**.

A solution of $Cu(NO_3)_2.3H_2O$ (50 mg) in ethanol (5 mL) was added to a cooled filtered solution of ligand 4'-(*p*-(1,4,8,11-tetraazacyclotetradec-1-yl)methylphenyl)-2,2':6',2"-terpyridine (cymt)**, 1.74**, (0.15 g), in ethanol (5 mL). The reaction mixture was heated at reflux for 1 h, and, upon cooling to room temperature, afforded a blue–green insoluble precipitate (0.11 g). The precipitate was suspended in ethanol–water (1:1, 5 mL), then the mixture was filtered after it was heated to reflux for 1 h. The solution was allowed to cool to room temperature overnight. The solution was kept in a refrigerator for about two months during which time blue crystals of **6.5** suitable for X-ray analysis were produced. No crystals of cymt, **1.74**, or its copper complex were produced in this way.

Appendix 1

X-Ray Crystallography

Tables A1.1-A1.5 list the crystal data and X-ray experimental details for the seventeen fully refined crystal structures discussed in this thesis. Throughout the text, selected bond lengths and angles are discussed and listed under the appropriate figures. Crystal data and structure refinement tables, atomic coordinates and equivalent isotropic displacement parameters tables, bond lengths and angles tables, anisotropic displacement parameters tables, hydrogen coordinates tables, and hydrogen bonds tables (Appendix 2); crystallographic information files (CIFs) (Appendix 3); and structure factor tables (Appendix 4) for all the structures discussed in this thesis are provided on the attached compact disc.

The X-ray data were collected on a Siemens P4 four circle diffractometer, using a Siemens SMART 1K CCD area detector and irradiating the sample with graphite monochromated $Mo_{K\alpha}$ (λ 0.71073 Å) radiation. The crystals were mounted 5.5 cm from the detector. The data were collected by the *SMART*[512] program and processed with the help of *SAINT*[513] to apply Lorentz and polarization corrections to the diffraction spots (three-dimensional integration). *SADABS*[514] was used to scale the diffractions if required. The structures were solved by direct and vector methods and refined using the *SHELXTL*[515] program. Hydrogen atoms were placed at calculated ideal positions and refined using a riding model. Hydrogen atoms in the structure **6.2**

(in Chapter 6) were located in a difference Fourier map. The O-H distances in that structure were constrained to 1.00(2) Å; with $U_{iso}(H)=1.2U_{eq}(O)$.

CCDC (Con Quest 1.7) was used for the search of the chemical structures.

Table A1.1. Crystal data and X-ray experimental data for **2.7 (A)**, **2.8 + 2.9 (B)**, **2.10 (C)**, and **2.21 (D)**.

Compound	$[Ni(tpt)(H_2O)_3]Cl_2.3H_2O$, **2.7 (A)**	$\{[Ni(tpt)Cl(H_2O)_2]Cl+[Ni(tpt)Cl_2(H_2O)]\}.4H_2O$, **2.8 + 2.9 (B)**	$[Ni(HTPT)Cl(H_2O)_2]Cl_2.2H_2O$, **2.10 (C)**	$[Ni(tpt)(H_2O)_3](NO_3)_2$, **2.12 (D)**
Formula	$C_{18}H_{24}Cl_2N_6NiO_6$	$C_{36}H_{38}Cl_4N_{12}Ni_2O_7$	$C_{18}H_{21}Cl_3N_6NiO_4$	$C_{18}H_{18}N_8NiO_9$
M	550.04	1010.00	550.47	549.11
Crystal system	Monoclinic	Triclinic	Monoclinic	Triclinic
Space group	*C*2/*c*	$P\bar{1}$	$P2_1/n$	$P\bar{1}$
a/Å	16.963(3)	13.0080(14)	12.4599(10)	7.4053(8)
b/Å	14.677(3)	13.3765(15)	14.5521(11)	13.3116(14)
c/Å	18.776(4)	13.9985(15)	13.3774(10)	13.8265(15)
α/°	90.00	85.3040(10)	90.00	63.4740(10)
β/°	95.05(3)	67.4600(2)	107.3680(10)	77.8480(10)
γ/°	90.00	69.0390(10)	90.00	84.5990(10)
V/Å^3	4656.5(16)	2096.5(4)	2315.0(3)	1192.2(2)
Z	8	2	4	2
T/K	293(2)	293(2)	169(2)	164(2)
μ/mm^{-1}	1.110	1.217	1.222	0.879
Reflections collected	16317	20130	28660	14726
Independent reflections	4811	8363	4739	4726
Observed	2948	5944	4428	3765
Parameters refined	336	586	321	327
R [I > 2σ(I)]	0.0625	0.0360	0.0331	0.0468
Rw [I > 2σ(I)]	0.1857	0.0772	0.0724	0.1208

Table A1.2. Crystal data and X-ray experimental data for **2.13 (E)**, **2.15 (F)**, **3.10**, and **3.11**.

Compound	$[Ni(tpt)_2](ClO_4)_2$, **2.13 (E)**	$[Ni_2(tpt)(EtOH)_2(NO3)_3(H_2O)](NO_3)$, **2.15 (F)**	$[Ru(tpm)(bpy)Cl](PF_6)$, **3.10**	$[Ru(tpm)(bpy)(H_2O)](ClO_4)_2$.MeOH, **3.11**
Formula	$C_{36}H_{24}Cl_2N_{12}NiO_8$	$C_{22}H_{26}N_{10}Ni_2O_{15}$	$C_{20}H_{18}ClF_6N_8PRu$	$C_{21}H_{24}Cl_2N_8O_{10}Ru$
M	882.28	787.95	651.91	720.45
Crystal system	Orthorhombic	Triclinic	Monoclinic	Monoclinic
Space group	*Pca*21	$P\bar{1}$	$P2_1/n$	$P2_1/c$
a/Å	15.886(3)	7.3769(3)	10.8239(9)	14.9764(17)
b/Å	11.519(2)	14.0212(4)	15.0829(13)	14.4923(18)
c/Å	21.636(4)	15.1672(5)	14.3141(12)	14.3878(16)
α/°	90.00	73.018	90	90
β/°	90.00	84.192(2)	97.314(2)	116.902(2)
γ/°	90.00	89.706(2)	90	90
V/Å^3	3959.3(13)	1492.19(9)	2317.8(3)	2784.8(6)
Z	4	2	4	4
T/K	163(2)	108(2)	88(2)	113(2)
μ/mm^{-1}	0.692	1.353	0.938	0.824
Reflections collected	40287	13017	18093	16052
Independent reflections	7833	5974	4720	5653
Observed	5836	4672	3805	4140
Parameters refined	532	457	334	382
R [I > 2σ(I)]	0.0378	0.0359	0.0274	0.0445
Rw [I > 2σ(I)]	0.0718	0.0834	0.0650	0.1078

Table A1.3. Crystal data and X-ray experimental data for **3.13**, **3.14**, **1.71**, and **4.2**.

Compound	[Ru(ttp)(bpy)Cl](PF$_6$), **3.13**	[Ru(ttp)(bpy)(bpe)](PF$_6$)$_2$.MeOH, **3.14**	pzt, **1.71**	[Ag$_2$(pzt)$_2$](BF$_4$)$_2$, **4.2**
Formula	$C_{32}H_{25}ClF_6N_5PRu$	$C_{45}H_{39}F_{12}N_7OP_2Ru$	$C_{33}H_{27}N_9O$	$C_{66}H_{54}Ag_2B_2F_8N_{18}O_2$
M	761.06	1084.84	565.64	1520.63
Crystal system	Monoclinic	Monoclinic	Orthorhombic	Monoclinic
Space group	*P*2$_1$/*c*	*P*2$_1$/*c*	*Pbca*	*P*2$_1$/*c*
a/Å	13.0766(15)	10.7421(10)	11.429(11)	8.5329(16)
b/Å	19.140(3)	21.633(2)	8.715(7)	22.108(5)
c/Å	12.2565(16)	19.3655(14)	55.97(5)	17.393(4)
α/°	90	90	90	90
β/°	100.049(2)	99.463(2)	90	96.534(3)
γ/°	90	90	90	90
V/Å^3	3020.6(7)	4438.9(7)	5575(9)	3259.9(11)
Z	4	4	8	2
T/K	93(2)	93(2)	191(2)	293(2)
μ/mm^{-1}	0.731	0.522	0.087	0.684
Reflections collected	21587	38264	14399	28177
Independent reflections	5207	9001	4801	6637
Observed	3790	7398	3327	2895
Parameters refined	481	807	388	480
R [I > 2σ(I)]	0.0356	0.0397	0.0643	0.0503
Rw [I > 2σ(I)]	0.0692	0.0933	0.1336	0.0835

Table A1.4. Crystal data and X-ray experimental data for **4.7**, **4.8**, **6.2**, and **6.4**.

Compound	$[Cu(pzt)(ONO_2)_2].CH_3CN$, **4.7**	$[Zn(pzt)(Cl)_2]$, **4.8**	$[Ru(tpt)_2](PF_6)_2$, **6.2**	$[Fe(tpt)Cl_2]_2(\mu\text{-}O).2(H_2O)$ **6.4**
Formula	$C_{35}H_{30}CuN_{12}O_7$	$C_{33}H_{30}Cl_2N_{10}OZn$	$C_{36}H_{24}F_{12}N_{12}P_2Ru$	$C_{36}H_{27}Cl_4Fe_2N_{12}O_3$
M	794.25	742.96	1015.68	928.19
Crystal system	Triclinic	Triclinic	Orthorhumbic	Monoclinic
Space group	$P\bar{1}$	$P\bar{1}$	*Pcca*	$P2_1/c$
a/Å	8.8307(7)	8.3507(15)	21.474(11)	10.8726(11)
b/Å	11.8013(9)	11.1461(15)	11.008(6)	15.8656(16)
c/Å	17.3963(13)	18.1240(15)	c=16.044(8)	22.747(2)
α/°	88.3580(10)	86.826(3)	90	90
β/°	79.6760(10)	82.396(2)	90	93.135(2)
γ/°	83.2590(10)	85.480(2)	90	90
V/Å^3	1771.2(2)	1665.2(5)	3793(3)	3918.0(7)
Z	2	2	4	4
T/K	168(2)	93(2)	85(2)	84(2)
μ/mm^{-1}	0.685	0.945	0.606	1.067
Reflections collected	20709	14016	21050	34106
Independent reflections	6267	6541	3864	7995
Observed	5586	5379	2935	6159
Parameters refined	509	443	287	525
R [I > 2σ(I)]	0.0357	0.0360	0.0584	0.0421
Rw [I > 2σ(I)]	0.0990	0.0869	0.1373	0.1212

Table A1.5. Crystal data and X-ray experimental data for **6.5**.

Compound	$[Cu(OH_2)_6](NO_3)_2$, **6.5**
Formula	$CuH_{12}N_2O_{12}$
M	295.66
Crystal system	Triclinic
Space group	$P\bar{1}$
a/Å	5.7404(8)
b/Å	7.6452(10)
c/Å	11.4655(15)
α°	106.428(2)
β°	98.399(2)
γ°	101.504(2)
V/Å3	461.84(11)
Z	2
T/K	93(2)
μ/mm^{-1}	2.432
Reflections collected	2917
Independent reflections	1556
Observed	1494
Parameters refined	172
R [I > 2σ(I)]	0.0343
Rw [I > 2σ(I)]	0.0952

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Appendix 2

- ***Crystal Data and Structure Refinement Tables***
- ***Atomic Coordinates and Equivalent Isotropic Displacement Parameters Tables***
- ***Selected Bond Lengths and Angles Tables***
- ***Bond Lengths and Angles Tables***
- ***Anisotropic Displacement Parameters Tables***
- ***Hydrogen Coordinates Tables***
- ***Hydrogen Bonds Tables***

Table A2.1. Crystal data and structure refinement for $[Ni(tpt)(H_2O)_3]Cl_2.3H_2O$, **2.7 (A).**

Identification code	**2.7 (A)**	
Empirical formula	C18 H24 Cl2 N6 Ni O6	
Formula weight	550.04	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 16.963(3) Å	α= 90°.
	b = 14.677(3) Å	β= 95.05(3)°.
	c = 18.776(4) Å	γ = 90°.
Volume	4656.5(16) $Å^3$	
Z	8	
Density (calculated)	1.569 Mg/m^3	
Absorption coefficient	1.110 mm^{-1}	
F(000)	2272	
Crystal size	0.75 x 0.45 x 0.20 mm^3	
Theta range for data collection	1.84 to 26.74°.	
Index ranges	-21<=h<=13, -13<=k<=18, -22<=l<=23	
Reflections collected	16317	
Independent reflections	4811 [R(int) = 0.0459]	
Completeness to theta = 26.74°	97.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8085 and 0.4899	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4811 / 26 / 336	
Goodness-of-fit on F^2	0.970	
Final R indices [I>2sigma(I)]	R1 = 0.0625, wR2 = 0.1857	
R indices (all data)	R1 = 0.0978, wR2 = 0.2004	
Largest diff. peak and hole	1.198 and -1.385 e.$Å^{-3}$	

Table A2.2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[Ni(tpt)(H_2O)_3]Cl_2.3H_2O$, **2.7 (A)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni	2144(1)	1419(1)	3362(1)	32(1)
N(1)	1407(2)	747(3)	2676(2)	31(1)
N(2)	451(2)	763(3)	1695(2)	31(1)
N(3)	967(2)	-650(2)	2185(2)	32(1)
N(4)	2382(2)	9(3)	3636(2)	34(1)
N(5)	1512(2)	2493(3)	2768(2)	33(1)
N(6)	-521(2)	-167(3)	744(2)	36(1)
O(1)	1346(2)	1472(2)	4129(2)	43(1)
O(2)	2991(2)	1480(2)	2648(2)	44(1)
O(3)	2981(3)	2076(3)	4051(2)	54(1)
C(1)	930(3)	1181(3)	2188(2)	31(1)
C(2)	502(3)	-157(3)	1705(2)	30(1)
C(3)	1408(3)	-166(3)	2659(3)	31(1)
C(4)	1950(3)	-595(3)	3223(3)	33(1)
C(5)	1988(3)	-1520(3)	3325(3)	38(1)
C(6)	2490(3)	-1868(3)	3889(3)	43(1)
C(7)	2926(3)	-1264(4)	4304(3)	46(1)
C(8)	2868(3)	-341(4)	4162(3)	39(1)
C(9)	986(3)	2188(3)	2228(2)	30(1)
C(10)	549(3)	2741(3)	1767(3)	34(1)
C(11)	638(3)	3683(3)	1859(3)	41(1)
C(12)	1161(3)	4003(3)	2405(3)	40(1)
C(13)	1583(3)	3396(3)	2837(3)	38(1)
C(14)	14(3)	-651(3)	1145(3)	34(1)
C(15)	135(3)	-1572(3)	1042(3)	50(2)
C(16)	-306(4)	-2003(4)	499(3)	61(2)
C(17)	-842(3)	-1505(4)	58(3)	50(1)
C(18)	-942(3)	-598(4)	214(3)	41(1)
Cl(1)	2481(1)	560(1)	1237(1)	49(1)
Cl(2)	4565(2)	1183(2)	3307(3)	205(2)
O(4)	4061(2)	1595(2)	5370(2)	39(1)
O(5)	2702(5)	3712(3)	4530(3)	107(2)

O(6)	5755(5)	407(9)	4151(5)	176(4)

Table A2.3. Selected bond lengths [Å] and angles [°] for $[Ni(tpt)(H_2O)_3]Cl_2.3H_2O$, **2.7 (A)**.

Ni-N(1)	1.977(4)
Ni-O(2)	2.051(4)
Ni-O(1)	2.063(4)
Ni-O(3)	2.073(4)
Ni-N(5)	2.160(4)
Ni-N(4)	2.162(4)
N(1)-Ni-O(2)	92.09(14)
N(1)-Ni-O(1)	93.28(15)
O(2)-Ni-O(1)	174.13(14)
N(1)-Ni-O(3)	176.00(16)
O(2)-Ni-O(3)	84.59(15)
O(1)-Ni-O(3)	90.13(16)
N(1)-Ni-N(5)	76.79(15)
O(2)-Ni-N(5)	88.60(14)
O(1)-Ni-N(5)	90.34(14)
O(3)-Ni-N(5)	105.31(16)
N(1)-Ni-N(4)	76.82(15)
O(2)-Ni-N(4)	94.08(14)
O(1)-Ni-N(4)	89.43(14)
O(3)-Ni-N(4)	101.14(16)
N(5)-Ni-N(4)	153.55(15)

Symmetry transformations used to generate equivalent atoms:

Table A2.4. Bond lengths [Å] and angles [°] for $[Ni(tpt)(H_2O)_3]Cl_2.3H_2O$, **2.7 (A)**.

Ni-N(1)	1.977(4)
Ni-O(2)	2.051(4)
Ni-O(1)	2.063(4)
Ni-O(3)	2.073(4)
Ni-N(5)	2.160(4)
Ni-N(4)	2.162(4)
N(1)-C(1)	1.331(6)
N(1)-C(3)	1.340(6)
N(2)-C(1)	1.328(6)
N(2)-C(2)	1.352(6)
N(3)-C(3)	1.319(6)
N(3)-C(2)	1.354(6)
N(4)-C(8)	1.333(6)
N(4)-C(4)	1.350(6)
N(5)-C(13)	1.337(6)
N(5)-C(9)	1.364(6)
N(6)-C(18)	1.332(6)
N(6)-C(14)	1.333(6)
O(1)-H(1A)	0.831(18)
O(1)-H(1B)	0.810(18)
O(2)-H(2A)	0.812(18)
O(2)-H(2B)	0.817(18)
O(3)-H(3A)	0.809(17)
O(3)-H(3B)	0.791(17)
C(1)-C(9)	1.482(6)
C(2)-C(14)	1.469(7)
C(3)-C(4)	1.480(7)
C(4)-C(5)	1.371(6)
C(5)-C(6)	1.396(7)
C(5)-H(5)	0.9300
C(6)-C(7)	1.356(7)
C(6)-H(6)	0.9300
C(7)-C(8)	1.383(7)
C(7)-H(7)	0.9300
C(8)-H(8)	0.9300
C(9)-C(10)	1.359(6)

C(10)-C(11)	1.400(6)
C(10)-H(10)	0.9300
C(11)-C(12)	1.377(7)
C(11)-H(11)	0.9300
C(12)-C(13)	1.365(7)
C(12)-H(12)	0.9300
C(13)-H(13)	0.9300
C(14)-C(15)	1.383(6)
C(15)-C(16)	1.367(8)
C(15)-H(15)	0.9300
C(16)-C(17)	1.383(8)
C(16)-H(16)	0.9300
C(17)-C(18)	1.378(7)
C(17)-H(17)	0.9300
C(18)-H(18)	0.9300
O(4)-H(4A)	0.903(14)
O(4)-H(4A)	0.903(14)
O(4)-H(4B)	0.830(14)
O(5)-H(5A)	0.86(2)
O(5)-H(5B)	0.86(2)
O(6)-H(6A)	0.85(2)
O(6)-H(6B)	0.83(2)

N(1)-Ni-O(2)	92.09(14)
N(1)-Ni-O(1)	93.28(15)
O(2)-Ni-O(1)	174.13(14)
N(1)-Ni-O(3)	176.00(16)
O(2)-Ni-O(3)	84.59(15)
O(1)-Ni-O(3)	90.13(16)
N(1)-Ni-N(5)	76.79(15)
O(2)-Ni-N(5)	88.60(14)
O(1)-Ni-N(5)	90.34(14)
O(3)-Ni-N(5)	105.31(16)
N(1)-Ni-N(4)	76.82(15)
O(2)-Ni-N(4)	94.08(14)
O(1)-Ni-N(4)	89.43(14)
O(3)-Ni-N(4)	101.14(16)
N(5)-Ni-N(4)	153.55(15)

C(1)-N(1)-C(3)	117.7(4)
C(1)-N(1)-Ni	121.4(3)
C(3)-N(1)-Ni	120.8(3)
C(1)-N(2)-C(2)	114.8(4)
C(3)-N(3)-C(2)	115.1(4)
C(8)-N(4)-C(4)	116.2(4)
C(8)-N(4)-Ni	129.5(3)
C(4)-N(4)-Ni	114.2(3)
C(13)-N(5)-C(9)	116.4(4)
C(13)-N(5)-Ni	129.6(3)
C(9)-N(5)-Ni	114.0(3)
C(18)-N(6)-C(14)	117.6(4)
Ni-O(1)-H(1A)	110.2(19)
Ni-O(1)-H(1B)	109.7(19)
H(1A)-O(1)-H(1B)	113(3)
Ni-O(2)-H(2A)	111.3(19)
Ni-O(2)-H(2B)	111.9(19)
H(2A)-O(2)-H(2B)	114(3)
Ni-O(3)-H(3A)	109.0(18)
Ni-O(3)-H(3B)	110.4(19)
H(3A)-O(3)-H(3B)	117(3)
N(2)-C(1)-N(1)	123.8(4)
N(2)-C(1)-C(9)	122.0(4)
N(1)-C(1)-C(9)	114.2(4)
N(2)-C(2)-N(3)	125.0(4)
N(2)-C(2)-C(14)	116.9(4)
N(3)-C(2)-C(14)	118.1(4)
N(3)-C(3)-N(1)	123.6(4)
N(3)-C(3)-C(4)	122.3(4)
N(1)-C(3)-C(4)	114.2(4)
N(4)-C(4)-C(5)	123.5(5)
N(4)-C(4)-C(3)	113.8(4)
C(5)-C(4)-C(3)	122.6(4)
C(4)-C(5)-C(6)	119.2(5)
C(4)-C(5)-H(5)	120.4
C(6)-C(5)-H(5)	120.4
C(7)-C(6)-C(5)	117.5(5)
C(7)-C(6)-H(6)	121.3

C(5)-C(6)-H(6)	121.3
C(6)-C(7)-C(8)	120.3(5)
C(6)-C(7)-H(7)	119.8
C(8)-C(7)-H(7)	119.8
N(4)-C(8)-C(7)	123.2(5)
N(4)-C(8)-H(8)	118.4
C(7)-C(8)-H(8)	118.4
C(10)-C(9)-N(5)	124.2(4)
C(10)-C(9)-C(1)	122.3(4)
N(5)-C(9)-C(1)	113.5(4)
C(9)-C(10)-C(11)	117.7(5)
C(9)-C(10)-H(10)	121.2
C(11)-C(10)-H(10)	121.2
C(12)-C(11)-C(10)	119.0(5)
C(12)-C(11)-H(11)	120.5
C(10)-C(11)-H(11)	120.5
C(13)-C(12)-C(11)	119.3(5)
C(13)-C(12)-H(12)	120.3
C(11)-C(12)-H(12)	120.3
N(5)-C(13)-C(12)	123.5(5)
N(5)-C(13)-H(13)	118.3
C(12)-C(13)-H(13)	118.3
N(6)-C(14)-C(15)	122.9(5)
N(6)-C(14)-C(2)	116.9(4)
C(15)-C(14)-C(2)	120.1(5)
C(16)-C(15)-C(14)	118.6(5)
C(16)-C(15)-H(15)	120.7
C(14)-C(15)-H(15)	120.7
C(15)-C(16)-C(17)	119.4(5)
C(15)-C(16)-H(16)	120.3
C(17)-C(16)-H(16)	120.3
C(18)-C(17)-C(16)	118.0(5)
C(18)-C(17)-H(17)	121.0
C(16)-C(17)-H(17)	121.0
N(6)-C(18)-C(17)	123.4(5)
N(6)-C(18)-H(18)	118.3
C(17)-C(18)-H(18)	118.3
H(4A)-O(4)-H(4B)	104(3)

H(4A)-O(4)-H(4B)	104(3)
H(5A)-O(5)-H(5B)	104(3)
H(6A)-O(6)-H(6B)	110(4)

Symmetry transformations used to generate equivalent atoms:

Table A2.5. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $[Ni(tpt)(H_2O)_3]Cl_2.3H_2O$, **2.7 (A)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	40(1)	27(1)	30(1)	-3(1)	-1(1)	-2(1)
N(1)	33(2)	30(2)	32(2)	0(2)	8(2)	-2(2)
N(2)	30(2)	32(2)	30(2)	-5(2)	2(2)	1(2)
N(3)	36(2)	26(2)	35(2)	-4(2)	5(2)	-1(2)
N(4)	40(2)	30(2)	30(2)	-6(2)	0(2)	0(2)
N(5)	38(2)	27(2)	34(2)	-1(2)	6(2)	-2(2)
N(6)	40(2)	37(2)	31(2)	-6(2)	3(2)	-7(2)
O(1)	57(2)	31(2)	42(2)	0(2)	9(2)	2(2)
O(2)	38(2)	54(2)	40(2)	-1(2)	2(2)	-9(2)
O(3)	70(3)	45(2)	47(2)	-4(2)	-3(2)	-8(2)
C(1)	32(2)	31(2)	30(3)	-1(2)	9(2)	2(2)
C(2)	32(2)	29(2)	31(3)	-6(2)	4(2)	-1(2)
C(3)	33(3)	25(2)	35(3)	-1(2)	9(2)	2(2)
C(4)	33(3)	31(2)	37(3)	-1(2)	11(2)	0(2)
C(5)	41(3)	30(2)	45(3)	-4(2)	8(2)	2(2)
C(6)	51(3)	33(3)	45(3)	3(2)	7(3)	9(2)
C(7)	54(3)	46(3)	38(3)	3(2)	-2(3)	14(3)
C(8)	42(3)	41(3)	33(3)	-5(2)	-3(2)	6(2)
C(9)	33(2)	26(2)	32(3)	-1(2)	7(2)	-3(2)
C(10)	35(3)	31(2)	38(3)	3(2)	7(2)	0(2)
C(11)	48(3)	32(3)	43(3)	4(2)	10(2)	3(2)
C(12)	48(3)	22(2)	50(3)	1(2)	10(3)	0(2)
C(13)	48(3)	29(2)	37(3)	-6(2)	5(2)	-8(2)
C(14)	35(3)	31(2)	37(3)	-6(2)	8(2)	-1(2)
C(15)	48(3)	39(3)	63(4)	-19(3)	-9(3)	9(2)
C(16)	61(4)	43(3)	76(5)	-30(3)	-8(3)	5(3)
C(17)	48(3)	51(3)	49(3)	-22(3)	2(3)	-6(3)
C(18)	41(3)	44(3)	35(3)	-8(2)	-1(2)	-4(2)
Cl(1)	51(1)	55(1)	41(1)	-7(1)	2(1)	3(1)
Cl(2)	104(2)	126(2)	372(6)	-74(3)	-59(3)	8(2)
O(4)	38(2)	35(2)	45(2)	-2(2)	7(2)	-5(2)
O(5)	207(7)	57(3)	51(3)	-3(2)	-28(4)	-4(4)
O(6)	114(6)	253(11)	172(8)	-60(8)	68(6)	-67(6)

Table A2.6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $[Ni(tpt)(H_2O)_3]Cl_2.3H_2O$, **2.7 (A)**.

	x	y	z	U(eq)
H(1A)	1120(20)	971(15)	4160(20)	65
H(1B)	1040(20)	1890(20)	4040(20)	65
H(2A)	3433(9)	1430(40)	2850(15)	67
H(2B)	2900(20)	1130(30)	2315(16)	67
H(3A)	2850(20)	2604(12)	4087(19)	30(14)
H(3B)	3060(20)	1800(20)	4410(14)	45
H(5)	1685	-1909	3022	46
H(6)	2523	-2491	3977	51
H(7)	3265	-1472	4685	55
H(8)	3184	56	4449	47
H(10)	203	2502	1403	41
H(11)	348	4086	1557	49
H(12)	1226	4626	2478	47
H(13)	1940	3623	3198	46
H(15)	509	-1891	1337	60
H(16)	-246	-2625	425	73
H(17)	-1127	-1776	-332	60
H(18)	-1323	-269	-65	49
H(4A)	4320(30)	1170(30)	5140(30)	59
H(4B)	4410(20)	1820(40)	5660(20)	59
H(5A)	2830(60)	4190(40)	4310(40)	161
H(5B)	2900(60)	3790(60)	4960(20)	161
H(6A)	5480(80)	0(80)	4340(70)	265
H(6B)	5630(90)	430(120)	3710(20)	265

Table A2.7. Hydrogen bonds for $[Ni(tpt)(H_2O)_3]Cl_2.3H_2O$, **2.7 (A)** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1B)...O(4)#1	0.810(18)	2.50(4)	3.086(5)	130(5)
O(2)-H(2A)...Cl(2)	0.812(18)	2.07(2)	2.874(5)	174(5)
O(2)-H(2B)...Cl(1)	0.817(18)	2.24(2)	3.031(4)	162(5)
O(3)-H(3A)...O(5)	0.809(17)	1.85(2)	2.621(7)	158(3)
O(3)-H(3B)...O(4)	0.791(17)	2.38(2)	3.031(6)	140(3)
O(3)-H(3B)...O(5)#1	0.791(17)	2.58(4)	3.213(8)	138(4)
O(5)-H(5A)...Cl(1)#2	0.86(2)	2.29(5)	3.075(5)	152(10)
O(5)-H(5B)...O(1)#1	0.86(2)	2.07(5)	2.883(7)	157(10)
O(6)-H(6A)...O(4)#3	0.85(2)	2.51(14)	3.081(14)	125(14)
O(6)-H(6B)...Cl(2)	0.83(2)	2.20(16)	2.706(13)	119(15)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1/2,-y+1/2,-z+1 #2 -x+1/2,y+1/2,-z+1/2

#3 -x+1,-y,-z+1

Table A2.8. Crystal data and structure refinement for {[Ni(tpt)Cl(H_2O)$_2$]Cl+[Ni(tpt)Cl_2(H_2O)]}.4H_2O, **2.8 + 2.9 (B)**.

Identification code	**2.8 + 2.9 (B)**	
Empirical formula	C36 H38 Cl4 N12 Ni2 O7	
Formula weight	1010.00	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 13.0080(14) Å	α= 85.3040(10)°.
	b = 13.3765(15) Å	β= 67.460(2)°.
	c = 13.9985(15) Å	γ = 69.0390(10)°.
Volume	2096.5(4) Å^3	
Z	2	
Density (calculated)	1.600 Mg/m^3	
Absorption coefficient	1.217 mm^{-1}	
F(000)	1036	
Crystal size	0.55 x 0.2 x 0.12 mm^3	
Theta range for data collection	1.63 to 26.40°.	
Index ranges	-11<=h<=16, -16<=k<=16, -13<=l<=17	
Reflections collected	20130	
Independent reflections	8363 [R(int) = 0.0367]	
Completeness to theta = 26.40°	97.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.864 and 0.723	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8363 / 5 / 586	
Goodness-of-fit on F^2	1.014	
Final R indices [I>2sigma(I)]	R1 = 0.0360, wR2 = 0.0772	
R indices (all data)	R1 = 0.0674, wR2 = 0.0856	
Largest diff. peak and hole	0.323 and -0.455 e.Å^{-3}	

Table A2.9. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for {[Ni(tpt)Cl(H_2O)$_2$]Cl+[Ni(tpt)Cl$_2$(H_2O)]}.4H_2O, **2.8 + 2.9 (B)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni(1)	2141(1)	5926(1)	2424(1)	22(1)
N(1)	3826(2)	4941(2)	2006(2)	21(1)
N(2)	5853(2)	4597(2)	1018(2)	20(1)
N(3)	5199(2)	3226(2)	2001(2)	21(1)
N(4)	1986(2)	4481(2)	3145(2)	22(1)
N(5)	3124(2)	6858(2)	1437(2)	22(1)
N(6)	8133(2)	3224(2)	379(2)	27(1)
Cl(1)	2221(1)	6609(1)	3970(1)	29(1)
Cl(2)	201(1)	7073(1)	2801(1)	37(1)
O(1)	2190(2)	5193(2)	1117(2)	29(1)
C(1)	4716(2)	5237(2)	1363(2)	19(1)
C(2)	6051(2)	3604(2)	1380(2)	19(1)
C(3)	4094(2)	3927(2)	2278(2)	19(1)
C(4)	3031(2)	3646(2)	2909(2)	21(1)
C(5)	3103(2)	2626(2)	3218(2)	29(1)
C(6)	2050(3)	2459(2)	3806(2)	35(1)
C(7)	984(2)	3304(2)	4071(2)	32(1)
C(8)	986(2)	4297(2)	3723(2)	26(1)
C(9)	4318(2)	6351(2)	1042(2)	20(1)
C(10)	5102(2)	6812(2)	380(2)	24(1)
C(11)	4632(3)	7862(2)	122(2)	30(1)
C(12)	3422(3)	8386(2)	521(2)	29(1)
C(13)	2690(2)	7866(2)	1174(2)	25(1)
C(14)	7305(2)	2863(2)	1065(2)	20(1)
C(15)	7585(2)	1859(2)	1455(2)	26(1)
C(16)	8763(2)	1197(2)	1151(2)	33(1)
C(17)	9625(3)	1563(2)	465(2)	38(1)
C(18)	9261(2)	2570(2)	96(2)	35(1)
Ni(1')	3569(1)	8725(1)	5038(1)	21(1)
N(1')	3222(2)	7492(2)	5804(2)	20(1)
N(2')	3939(2)	5789(2)	6393(2)	23(1)
N(3')	1832(2)	6744(2)	6917(2)	22(1)

N(4')	1642(2)	9309(2)	5784(2)	23(1)
N(5')	5314(2)	7544(2)	4725(2)	23(1)
N(6')	1438(2)	5098(2)	8118(2)	30(1)
Cl(1')	3843(1)	9463(1)	6374(1)	30(1)
O(1')	3557(2)	8156(2)	3700(2)	39(1)
O(2')	3821(2)	10026(2)	4229(1)	27(1)
C(1')	4101(2)	6613(2)	5837(2)	22(1)
C(2')	2785(2)	5891(2)	6921(2)	21(1)
C(3')	2106(2)	7529(2)	6356(2)	21(1)
C(4')	1180(2)	8576(2)	6324(2)	22(1)
C(5')	-15(2)	8783(2)	6845(2)	27(1)
C(6')	-809(2)	9802(2)	6825(2)	33(1)
C(7')	-353(2)	10570(2)	6297(2)	33(1)
C(8')	865(2)	10287(2)	5791(2)	27(1)
C(9')	5310(2)	6626(2)	5215(2)	21(1)
C(10')	6325(2)	5788(2)	5160(2)	25(1)
C(11')	7417(2)	5878(2)	4569(2)	30(1)
C(12')	7444(2)	6803(2)	4075(2)	33(1)
C(13')	6381(2)	7615(2)	4165(2)	29(1)
C(14')	2580(2)	4990(2)	7558(2)	23(1)
C(15')	3533(2)	4082(2)	7557(2)	25(1)
C(16')	3309(3)	3252(2)	8162(2)	31(1)
C(17')	2138(3)	3376(2)	8753(2)	34(1)
C(18')	1250(3)	4296(2)	8707(2)	40(1)
Cl(10)	4176(1)	9761(1)	1995(1)	43(1)
O(10)	2428(2)	1581(2)	1150(2)	51(1)
O(11)	1610(2)	1129(2)	7996(2)	56(1)
O(12)	3034(2)	657(2)	9199(2)	53(1)
O(13)	1028(2)	3794(2)	1462(2)	47(1)

Table A2.10. Selected bond lengths [Å] and angles [°] for {[Ni(tpt)Cl(H_2O)$_2$]Cl+[Ni(tpt)Cl$_2$(H_2O)]}.4H_2O, **2.8 + 2.9 (B)**.

Ni(1)-N(1)	1.989(2)
Ni(1)-O(1)	2.1148(19)
Ni(1)-N(4)	2.144(2)
Ni(1)-N(5)	2.145(2)
Ni(1)-Cl(2)	2.3113(8)
Ni(1)-Cl(1)	2.4695(8)
Ni(1')-N(1')	1.987(2)
Ni(1')-O(2')	2.0535(18)
Ni(1')-O(1')	2.086(2)
Ni(1')-N(5')	2.151(2)
Ni(1')-N(4')	2.171(2)
Ni(1')-Cl(1')	2.3801(8)
N(1)-Ni(1)-O(1)	84.64(8)
N(1)-Ni(1)-N(4)	76.84(8)
O(1)-Ni(1)-N(4)	84.49(8)
N(1)-Ni(1)-N(5)	76.67(8)
O(1)-Ni(1)-N(5)	90.54(8)
N(4)-Ni(1)-N(5)	153.39(8)
N(1)-Ni(1)-Cl(2)	176.46(6)
O(1)-Ni(1)-Cl(2)	92.35(6)
N(4)-Ni(1)-Cl(2)	104.76(6)
N(5)-Ni(1)-Cl(2)	101.55(6)
N(1)-Ni(1)-Cl(1)	90.64(6)
O(1)-Ni(1)-Cl(1)	174.35(6)
N(4)-Ni(1)-Cl(1)	91.42(6)
N(5)-Ni(1)-Cl(1)	91.39(6)
Cl(2)-Ni(1)-Cl(1)	92.47(3)
N(1')-Ni(1')-O(2')	176.06(8)
N(1')-Ni(1')-O(1')	91.53(8)
O(2')-Ni(1')-O(1')	86.42(8)
N(1')-Ni(1')-N(5')	77.05(8)
O(2')-Ni(1')-N(5')	106.29(8)
O(1')-Ni(1')-N(5')	89.73(9)
N(1')-Ni(1')-N(4')	76.14(8)

O(2')-Ni(1')-N(4')	100.58(8)
O(1')-Ni(1')-N(4')	92.82(9)
N(5')-Ni(1')-N(4')	153.12(8)
N(1')-Ni(1')-Cl(1')	96.01(6)
O(2')-Ni(1')-Cl(1')	86.37(6)
O(1')-Ni(1')-Cl(1')	170.55(6)
N(5')-Ni(1')-Cl(1')	86.43(6)
N(4')-Ni(1')-Cl(1')	94.51(6)

Symmetry transformations used to generate equivalent atoms:

Table A2.11. Bond lengths [Å] and angles [°] for {[Ni(tpt)Cl(H_2O)$_2$]Cl+[Ni(tpt)Cl$_2$(H_2O)]}.4H_2O, **2.8 + 2.9 (B)**.

Ni(1)-N(1)	1.989(2)
Ni(1)-O(1)	2.1148(19)
Ni(1)-N(4)	2.144(2)
Ni(1)-N(5)	2.145(2)
Ni(1)-Cl(2)	2.3113(8)
Ni(1)-Cl(1)	2.4695(8)
N(1)-C(1)	1.333(3)
N(1)-C(3)	1.335(3)
N(2)-C(1)	1.330(3)
N(2)-C(2)	1.354(3)
N(3)-C(3)	1.328(3)
N(3)-C(2)	1.353(3)
N(4)-C(8)	1.341(3)
N(4)-C(4)	1.354(3)
N(5)-C(13)	1.344(3)
N(5)-C(9)	1.351(3)
N(6)-C(18)	1.329(3)
N(6)-C(14)	1.348(3)
O(1)-H(1A)	0.8200
O(1)-H(1B)	0.77(3)
C(1)-C(9)	1.485(3)
C(2)-C(14)	1.483(3)
C(3)-C(4)	1.490(3)
C(4)-C(5)	1.378(3)
C(5)-C(6)	1.389(4)
C(5)-H(5)	0.9300
C(6)-C(7)	1.375(4)
C(6)-H(6)	0.9300
C(7)-C(8)	1.378(4)
C(7)-H(7)	0.9300
C(8)-H(8)	0.9300
C(9)-C(10)	1.383(3)
C(10)-C(11)	1.396(4)
C(10)-H(10)	0.9300
C(11)-C(12)	1.372(4)

C(11)-H(11)	0.9300
C(12)-C(13)	1.389(4)
C(12)-H(12)	0.9300
C(13)-H(13)	0.9300
C(14)-C(15)	1.385(3)
C(15)-C(16)	1.380(4)
C(15)-H(15)	0.9300
C(16)-C(17)	1.381(4)
C(16)-H(16)	0.9300
C(17)-C(18)	1.386(4)
C(17)-H(17)	0.9300
C(18)-H(18)	0.9300
Ni(1')-N(1')	1.987(2)
Ni(1')-O(2')	2.0535(18)
Ni(1')-O(1')	2.086(2)
Ni(1')-N(5')	2.151(2)
Ni(1')-N(4')	2.171(2)
Ni(1')-Cl(1')	2.3801(8)
N(1')-C(1')	1.327(3)
N(1')-C(3')	1.342(3)
N(2')-C(1')	1.330(3)
N(2')-C(2')	1.356(3)
N(3')-C(3')	1.329(3)
N(3')-C(2')	1.353(3)
N(4')-C(8')	1.335(3)
N(4')-C(4')	1.359(3)
N(5')-C(13')	1.341(3)
N(5')-C(9')	1.358(3)
N(6')-C(18')	1.333(3)
N(6')-C(14')	1.348(3)
O(1')-H(1A')	0.8200
O(1')-H(1B')	0.79(3)
O(2')-H(2A')	0.8200
O(2')-H(2B')	0.773(17)
C(1')-C(9')	1.482(3)
C(2')-C(14')	1.479(3)
C(3')-C(4')	1.495(3)
C(4')-C(5')	1.371(3)

C(5')-C(6')	1.392(4)
C(5')-H(5')	0.9300
C(6')-C(7')	1.388(4)
C(6')-H(6')	0.9300
C(7')-C(8')	1.382(4)
C(7')-H(7')	0.9300
C(8')-H(8')	0.9300
C(9')-C(10')	1.372(3)
C(10')-C(11')	1.386(4)
C(10')-H(10')	0.9300
C(11')-C(12')	1.372(4)
C(11')-H(11')	0.9300
C(12')-C(13')	1.385(4)
C(12')-H(12')	0.9300
C(13')-H(13')	0.9300
C(14')-C(15')	1.391(3)
C(15')-C(16')	1.386(4)
C(15')-H(15')	0.9300
C(16')-C(17')	1.381(4)
C(16')-H(16')	0.9300
C(17')-C(18')	1.372(4)
C(17')-H(17')	0.9300
C(18')-H(18')	0.9300
O(10)-H(10A)	0.737(17)
O(10)-H(10B)	0.733(17)
O(11)-H(11A)	0.87(4)
O(11)-H(11B)	0.82(4)
O(12)-H(12A)	0.84(4)
O(12)-H(12B)	0.79(4)
O(13)-H(13A)	0.76(4)
O(13)-H(13B)	0.86(4)

N(1)-Ni(1)-O(1)	84.64(8)
N(1)-Ni(1)-N(4)	76.84(8)
O(1)-Ni(1)-N(4)	84.49(8)
N(1)-Ni(1)-N(5)	76.67(8)
O(1)-Ni(1)-N(5)	90.54(8)
N(4)-Ni(1)-N(5)	153.39(8)

N(1)-Ni(1)-Cl(2)	176.46(6)
O(1)-Ni(1)-Cl(2)	92.35(6)
N(4)-Ni(1)-Cl(2)	104.76(6)
N(5)-Ni(1)-Cl(2)	101.55(6)
N(1)-Ni(1)-Cl(1)	90.64(6)
O(1)-Ni(1)-Cl(1)	174.35(6)
N(4)-Ni(1)-Cl(1)	91.42(6)
N(5)-Ni(1)-Cl(1)	91.39(6)
Cl(2)-Ni(1)-Cl(1)	92.47(3)
C(1)-N(1)-C(3)	117.7(2)
C(1)-N(1)-Ni(1)	121.06(17)
C(3)-N(1)-Ni(1)	120.96(17)
C(1)-N(2)-C(2)	114.6(2)
C(3)-N(3)-C(2)	114.7(2)
C(8)-N(4)-C(4)	117.6(2)
C(8)-N(4)-Ni(1)	127.67(18)
C(4)-N(4)-Ni(1)	114.53(16)
C(13)-N(5)-C(9)	117.6(2)
C(13)-N(5)-Ni(1)	127.65(17)
C(9)-N(5)-Ni(1)	114.74(16)
C(18)-N(6)-C(14)	116.7(2)
Ni(1)-O(1)-H(1A)	109.5
Ni(1)-O(1)-H(1B)	117(2)
H(1A)-O(1)-H(1B)	116.4
N(2)-C(1)-N(1)	123.8(2)
N(2)-C(1)-C(9)	122.6(2)
N(1)-C(1)-C(9)	113.6(2)
N(3)-C(2)-N(2)	125.4(2)
N(3)-C(2)-C(14)	117.1(2)
N(2)-C(2)-C(14)	117.5(2)
N(3)-C(3)-N(1)	123.7(2)
N(3)-C(3)-C(4)	122.8(2)
N(1)-C(3)-C(4)	113.5(2)
N(4)-C(4)-C(5)	123.1(2)
N(4)-C(4)-C(3)	113.8(2)
C(5)-C(4)-C(3)	123.0(2)
C(4)-C(5)-C(6)	117.9(3)
C(4)-C(5)-H(5)	121.1

C(6)-C(5)-H(5)	121.1
C(7)-C(6)-C(5)	119.7(3)
C(7)-C(6)-H(6)	120.1
C(5)-C(6)-H(6)	120.1
C(6)-C(7)-C(8)	118.8(3)
C(6)-C(7)-H(7)	120.6
C(8)-C(7)-H(7)	120.6
N(4)-C(8)-C(7)	122.8(2)
N(4)-C(8)-H(8)	118.6
C(7)-C(8)-H(8)	118.6
N(5)-C(9)-C(10)	123.4(2)
N(5)-C(9)-C(1)	113.9(2)
C(10)-C(9)-C(1)	122.7(2)
C(9)-C(10)-C(11)	118.0(2)
C(9)-C(10)-H(10)	121.0
C(11)-C(10)-H(10)	121.0
C(12)-C(11)-C(10)	119.1(2)
C(12)-C(11)-H(11)	120.5
C(10)-C(11)-H(11)	120.5
C(11)-C(12)-C(13)	119.6(2)
C(11)-C(12)-H(12)	120.2
C(13)-C(12)-H(12)	120.2
N(5)-C(13)-C(12)	122.3(2)
N(5)-C(13)-H(13)	118.9
C(12)-C(13)-H(13)	118.9
N(6)-C(14)-C(15)	123.0(2)
N(6)-C(14)-C(2)	116.1(2)
C(15)-C(14)-C(2)	120.9(2)
C(16)-C(15)-C(14)	119.2(2)
C(16)-C(15)-H(15)	120.4
C(14)-C(15)-H(15)	120.4
C(15)-C(16)-C(17)	118.6(3)
C(15)-C(16)-H(16)	120.7
C(17)-C(16)-H(16)	120.7
C(16)-C(17)-C(18)	118.3(3)
C(16)-C(17)-H(17)	120.8
C(18)-C(17)-H(17)	120.8
N(6)-C(18)-C(17)	124.3(3)

N(6)-C(18)-H(18)	117.9
C(17)-C(18)-H(18)	117.9
N(1')-Ni(1')-O(2')	176.06(8)
N(1')-Ni(1')-O(1')	91.53(8)
O(2')-Ni(1')-O(1')	86.42(8)
N(1')-Ni(1')-N(5')	77.05(8)
O(2')-Ni(1')-N(5')	106.29(8)
O(1')-Ni(1')-N(5')	89.73(9)
N(1')-Ni(1')-N(4')	76.14(8)
O(2')-Ni(1')-N(4')	100.58(8)
O(1')-Ni(1')-N(4')	92.82(9)
N(5')-Ni(1')-N(4')	153.12(8)
N(1')-Ni(1')-Cl(1')	96.01(6)
O(2')-Ni(1')-Cl(1')	86.37(6)
O(1')-Ni(1')-Cl(1')	170.55(6)
N(5')-Ni(1')-Cl(1')	86.43(6)
N(4')-Ni(1')-Cl(1')	94.51(6)
C(1')-N(1')-C(3')	117.2(2)
C(1')-N(1')-Ni(1')	120.52(17)
C(3')-N(1')-Ni(1')	122.16(16)
C(1')-N(2')-C(2')	114.8(2)
C(3')-N(3')-C(2')	114.3(2)
C(8')-N(4')-C(4')	116.6(2)
C(8')-N(4')-Ni(1')	128.59(18)
C(4')-N(4')-Ni(1')	114.82(16)
C(13')-N(5')-C(9')	116.8(2)
C(13')-N(5')-Ni(1')	129.10(18)
C(9')-N(5')-Ni(1')	113.97(16)
C(18')-N(6')-C(14')	116.8(2)
Ni(1')-O(1')-H(1A')	109.5
Ni(1')-O(1')-H(1B')	127(3)
H(1A')-O(1')-H(1B')	120.6
Ni(1')-O(2')-H(2A')	109.5
Ni(1')-O(2')-H(2B')	115(2)
H(2A')-O(2')-H(2B')	109.3
N(1')-C(1')-N(2')	124.0(2)
N(1')-C(1')-C(9')	114.5(2)
N(2')-C(1')-C(9')	121.5(2)

N(3')-C(2')-N(2')	125.3(2)
N(3')-C(2')-C(14')	118.8(2)
N(2')-C(2')-C(14')	115.9(2)
N(3')-C(3')-N(1')	124.3(2)
N(3')-C(3')-C(4')	122.4(2)
N(1')-C(3')-C(4')	113.3(2)
N(4')-C(4')-C(5')	123.9(2)
N(4')-C(4')-C(3')	113.5(2)
C(5')-C(4')-C(3')	122.5(2)
C(4')-C(5')-C(6')	118.5(3)
C(4')-C(5')-H(5')	120.7
C(6')-C(5')-H(5')	120.7
C(7')-C(6')-C(5')	118.4(3)
C(7')-C(6')-H(6')	120.8
C(5')-C(6')-H(6')	120.8
C(8')-C(7')-C(6')	119.1(3)
C(8')-C(7')-H(7')	120.5
C(6')-C(7')-H(7')	120.5
N(4')-C(8')-C(7')	123.5(3)
N(4')-C(8')-H(8')	118.2
C(7')-C(8')-H(8')	118.2
N(5')-C(9')-C(10')	123.5(2)
N(5')-C(9')-C(1')	113.8(2)
C(10')-C(9')-C(1')	122.6(2)
C(9')-C(10')-C(11')	118.5(3)
C(9')-C(10')-H(10')	120.7
C(11')-C(10')-H(10')	120.7
C(12')-C(11')-C(10')	119.0(3)
C(12')-C(11')-H(11')	120.5
C(10')-C(11')-H(11')	120.5
C(11')-C(12')-C(13')	119.2(3)
C(11')-C(12')-H(12')	120.4
C(13')-C(12')-H(12')	120.4
N(5')-C(13')-C(12')	122.9(3)
N(5')-C(13')-H(13')	118.6
C(12')-C(13')-H(13')	118.6
N(6')-C(14')-C(15')	122.7(2)
N(6')-C(14')-C(2')	116.6(2)

C(15')-C(14')-C(2')	120.7(2)
C(16')-C(15')-C(14')	119.2(3)
C(16')-C(15')-H(15')	120.4
C(14')-C(15')-H(15')	120.4
C(17')-C(16')-C(15')	118.0(3)
C(17')-C(16')-H(16')	121.0
C(15')-C(16')-H(16')	121.0
C(18')-C(17')-C(16')	119.2(3)
C(18')-C(17')-H(17')	120.4
C(16')-C(17')-H(17')	120.4
N(6')-C(18')-C(17')	124.1(3)
N(6')-C(18')-H(18')	117.9
C(17')-C(18')-H(18')	117.9
H(10A)-O(10)-H(10B)	109(3)
H(11A)-O(11)-H(11B)	100(3)
H(12A)-O(12)-H(12B)	94(4)
H(13A)-O(13)-H(13B)	110(4)

Symmetry transformations used to generate equivalent atoms:

Table A2.12. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for {[Ni(tpt)Cl(H_2O)$_2$]Cl+[Ni(tpt)Cl$_2$(H_2O)]}.4H_2O, **2.8 + 2.9 (B)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1)	17(1)	19(1)	26(1)	5(1)	-5(1)	-8(1)
N(1)	19(1)	19(1)	23(1)	4(1)	-6(1)	-8(1)
N(2)	18(1)	22(1)	20(1)	5(1)	-4(1)	-10(1)
N(3)	20(1)	19(1)	21(1)	4(1)	-5(1)	-8(1)
N(4)	19(1)	24(1)	22(1)	3(1)	-5(1)	-9(1)
N(5)	22(1)	19(1)	25(1)	3(1)	-9(1)	-8(1)
N(6)	21(1)	25(1)	32(1)	5(1)	-5(1)	-10(1)
Cl(1)	35(1)	24(1)	32(1)	4(1)	-14(1)	-13(1)
Cl(2)	18(1)	34(1)	51(1)	7(1)	-9(1)	-7(1)
O(1)	34(1)	28(1)	29(1)	10(1)	-11(1)	-18(1)
C(1)	22(1)	22(1)	17(1)	5(1)	-7(1)	-12(1)
C(2)	19(1)	21(1)	17(1)	-2(1)	-6(1)	-8(1)
C(3)	20(1)	19(1)	18(1)	2(1)	-6(1)	-8(1)
C(4)	21(1)	21(1)	18(1)	2(1)	-4(1)	-10(1)
C(5)	28(2)	21(2)	33(2)	7(1)	-6(1)	-11(1)
C(6)	34(2)	27(2)	41(2)	14(1)	-7(2)	-18(1)
C(7)	25(2)	35(2)	36(2)	7(1)	-6(1)	-19(1)
C(8)	19(1)	29(2)	26(2)	2(1)	-3(1)	-10(1)
C(9)	21(1)	20(1)	19(1)	2(1)	-6(1)	-9(1)
C(10)	25(2)	21(1)	26(2)	4(1)	-8(1)	-12(1)
C(11)	39(2)	30(2)	25(2)	9(1)	-9(1)	-23(1)
C(12)	40(2)	18(1)	32(2)	7(1)	-17(1)	-11(1)
C(13)	27(2)	20(1)	30(2)	6(1)	-13(1)	-9(1)
C(14)	20(1)	23(1)	18(1)	1(1)	-6(1)	-9(1)
C(15)	24(2)	27(2)	23(1)	5(1)	-5(1)	-11(1)
C(16)	31(2)	25(2)	33(2)	7(1)	-10(1)	-5(1)
C(17)	20(2)	34(2)	47(2)	3(2)	-8(1)	-1(1)
C(18)	21(2)	35(2)	40(2)	8(1)	-4(1)	-12(1)
Ni(1')	20(1)	19(1)	24(1)	5(1)	-7(1)	-10(1)
N(1')	18(1)	17(1)	24(1)	3(1)	-7(1)	-8(1)
N(2')	22(1)	19(1)	26(1)	3(1)	-7(1)	-10(1)
N(3')	19(1)	19(1)	26(1)	3(1)	-7(1)	-8(1)

N(4')	22(1)	22(1)	26(1)	7(1)	-10(1)	-11(1)
N(5')	19(1)	23(1)	25(1)	1(1)	-5(1)	-10(1)
N(6')	29(1)	23(1)	34(1)	8(1)	-6(1)	-12(1)
Cl(1')	32(1)	32(1)	28(1)	3(1)	-8(1)	-16(1)
O(1')	57(2)	41(1)	34(1)	11(1)	-21(1)	-36(1)
O(2')	30(1)	26(1)	24(1)	7(1)	-11(1)	-12(1)
C(1')	22(1)	23(1)	21(1)	1(1)	-7(1)	-9(1)
C(2')	23(1)	18(1)	24(1)	-1(1)	-8(1)	-9(1)
C(3')	18(1)	20(1)	25(1)	2(1)	-8(1)	-9(1)
C(4')	20(1)	20(1)	26(2)	5(1)	-9(1)	-7(1)
C(5')	21(2)	26(2)	36(2)	6(1)	-10(1)	-11(1)
C(6')	20(2)	31(2)	42(2)	3(1)	-10(1)	-5(1)
C(7')	27(2)	23(2)	44(2)	6(1)	-15(1)	-4(1)
C(8')	29(2)	21(2)	33(2)	8(1)	-13(1)	-10(1)
C(9')	20(1)	23(1)	22(1)	1(1)	-7(1)	-10(1)
C(10')	22(2)	27(2)	26(2)	2(1)	-9(1)	-8(1)
C(11')	19(1)	35(2)	33(2)	-1(1)	-8(1)	-7(1)
C(12')	19(2)	44(2)	36(2)	-2(1)	-4(1)	-18(1)
C(13')	27(2)	32(2)	28(2)	1(1)	-4(1)	-17(1)
C(14')	26(2)	19(1)	25(1)	3(1)	-8(1)	-11(1)
C(15')	25(2)	24(2)	29(2)	3(1)	-13(1)	-10(1)
C(16')	38(2)	20(2)	37(2)	6(1)	-19(1)	-8(1)
C(17')	42(2)	25(2)	38(2)	14(1)	-14(2)	-17(1)
C(18')	36(2)	34(2)	44(2)	14(2)	-6(2)	-19(2)
Cl(10)	49(1)	49(1)	36(1)	7(1)	-19(1)	-21(1)
O(10)	52(2)	49(2)	40(1)	-1(1)	-13(1)	-7(1)
O(11)	50(2)	51(2)	46(2)	4(1)	-16(1)	4(1)
O(12)	55(2)	49(2)	51(2)	11(1)	-22(1)	-14(1)
O(13)	34(1)	34(1)	72(2)	8(1)	-16(1)	-18(1)

Table A2.13. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for {[Ni(tpt)Cl(H_2O)$_2$]Cl+[Ni(tpt)Cl$_2$(H_2O)]}.4H_2O, **2.8 + 2.9 (B)**.

	x	y	z	U(eq)
H(1A)	2121	5635	683	43
H(1B)	1820(30)	4830(20)	1220(20)	43
H(5)	3834	2068	3039	35
H(6)	2066	1779	4020	42
H(7)	275	3207	4479	38
H(8)	262	4864	3895	31
H(10)	5918	6434	115	28
H(11)	5133	8202	-314	36
H(12)	3094	9085	355	35
H(13)	1870	8228	1439	30
H(15)	6987	1633	1917	31
H(16)	8973	519	1402	39
H(17)	10428	1144	255	45
H(18)	9844	2803	-380	41
H(1A')	3689	8567	3239	58
H(1B')	3280(30)	7730(30)	3660(30)	58
H(2A')	4378	10134	4285	40
H(2B')	3930(30)	9980(20)	3648(14)	40
H(5')	-289	8253	7203	33
H(6')	-1626	9966	7158	39
H(7')	-859	11263	6283	39
H(8')	1160	10807	5438	33
H(10')	6282	5174	5510	30
H(11')	8120	5320	4509	36
H(12')	8167	6884	3684	40
H(13')	6409	8237	3823	34
H(15')	4311	4032	7157	30
H(16')	3928	2631	8169	37
H(17')	1955	2841	9176	41
H(18')	467	4365	9112	47
H(10A)	2830(30)	1190(30)	1370(30)	70
H(11A)	1080(30)	1590(30)	7790(30)	70

H(12A)	3710(30)	600(30)	8770(30)	70
H(13A)	360(30)	3900(30)	1690(30)	70
H(10B)	2580(30)	1390(30)	620(17)	70
H(11B)	2090(30)	800(30)	7440(30)	70
H(12B)	2730(30)	800(30)	8790(30)	70
H(13B)	1420(30)	3110(30)	1380(30)	70

Table A2.14. Hydrogen bonds for {[Ni(tpt)Cl(H_2O)$_2$]Cl+[Ni(tpt)Cl$_2$(H_2O)]}.4H_2O, **2.8 + 2.9 (B)** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1A)...N(6)#1	0.82	2.07	2.891(3)	177.5
O(1)-H(1B)...O(13)	0.77(3)	1.94(3)	2.708(3)	174(3)
O(1')-H(1A')...Cl(10)	0.82	2.33	3.143(2)	174.9
O(1')-H(1B')...Cl(1)	0.79(3)	2.29(3)	3.063(2)	166(3)
O(2')-H(2A')...Cl(1')#2	0.82	2.38	3.137(2)	153.5
O(2')-H(2B')...Cl(10)	0.773(17)	2.241(17)	3.012(2)	175(3)
O(10)-H(10A)...Cl(10)#3	0.737(17)	2.447(18)	3.182(3)	174(4)
O(10)-H(10B)...O(12)#4	0.733(17)	2.068(19)	2.796(4)	173(4)
O(11)-H(11A)...Cl(2)#5	0.87(4)	2.31(4)	3.178(3)	174(3)
O(11)-H(11B)...Cl(1')#3	0.82(4)	2.35(4)	3.115(3)	155(3)
O(12)-H(12A)...Cl(10)#6	0.84(4)	2.41(4)	3.213(3)	161(4)
O(12)-H(12B)...O(11)	0.79(4)	2.07(4)	2.837(4)	167(4)
O(13)-H(13A)...N(6')#5	0.76(4)	2.16(4)	2.851(3)	151(4)
O(13)-H(13B)...O(10)	0.86(4)	1.97(4)	2.831(3)	175(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z #2 -x+1,-y+2,-z+1 #3 x,y-1,z

#4 x,y,z-1 #5 -x,-y+1,-z+1 #6 -x+1,-y+1,-z+1

Table A2.15. Crystal data and structure refinement for $[Ni(Htpt)Cl(H_2O)_2]Cl_2.2H_2O$, **2.10 (C)**.

Identification code	**2.10 (C)**	
Empirical formula	C18 H21 Cl3 N6 Ni O4	
Formula weight	550.47	
Temperature	169(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	p 21/n	
Unit cell dimensions	a = 12.4599(10) Å	α= 90°.
	b = 14.5521(11) Å	β= 107.3680(10)°.
	c = 13.3774(10) Å	γ = 90°.
Volume	2315.0(3) $Å^3$	
Z	4	
Density (calculated)	1.579 Mg/m^3	
Absorption coefficient	1.222 mm^{-1}	
F(000)	1128	
Crystal size	0.80 x 0.43 x 0.25 mm^3	
Theta range for data collection	1.96 to 26.45°.	
Index ranges	-15<=h<=15, -18<=k<=17, -16<=l<=16	
Reflections collected	28660	
Independent reflections	4739 [R(int) = 0.0284]	
Completeness to theta = 26.45°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.73480 and 0.43262	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4739 / 12 / 321	
Goodness-of-fit on F^2	1.204	
Final R indices [I>2sigma(I)]	R1 = 0.0331, wR2 = 0.0724	
R indices (all data)	R1 = 0.0366, wR2 = 0.0738	
Largest diff. peak and hole	0.299 and -0.512 e.$Å^{-3}$	

Table A2.16. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[Ni(Htpt)Cl(H_2O)_2]Cl_2.2H_2O$, **2.10 (C)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni	5570(1)	8419(1)	8872(1)	16(1)
Cl(1)	4782(1)	9851(1)	8353(1)	24(1)
O(1)	5925(1)	8349(1)	7448(1)	21(1)
O(2)	4965(2)	8470(1)	10160(1)	28(1)
N(1)	6362(1)	7217(1)	9334(1)	16(1)
N(2)	8018(1)	6407(1)	10179(1)	17(1)
N(3)	6299(1)	5603(1)	9324(1)	17(1)
N(4)	4267(1)	7410(1)	8279(1)	17(1)
N(5)	7279(1)	8821(1)	9735(1)	19(1)
N(6)	7350(2)	3983(1)	9811(1)	20(1)
C(1)	7455(2)	7187(1)	9876(2)	16(1)
C(2)	7396(2)	5648(1)	9873(2)	17(1)
C(3)	5814(2)	6424(1)	9079(2)	15(1)
C(4)	4608(2)	6522(1)	8489(2)	16(1)
C(5)	3899(2)	5772(2)	8197(2)	20(1)
C(6)	2774(2)	5937(2)	7662(2)	23(1)
C(7)	2409(2)	6841(2)	7456(2)	24(1)
C(8)	3177(2)	7557(2)	7771(2)	21(1)
C(9)	7988(2)	8107(1)	10116(2)	18(1)
C(10)	9110(2)	8218(1)	10697(2)	19(1)
C(11)	9524(2)	9109(2)	10891(2)	24(1)
C(12)	8814(2)	9844(2)	10508(2)	26(1)
C(13)	7696(2)	9675(2)	9928(2)	23(1)
C(14)	7973(2)	4749(1)	10158(2)	18(1)
C(15)	9089(2)	4660(2)	10740(2)	21(1)
C(16)	9558(2)	3780(2)	10937(2)	28(1)
C(17)	8902(2)	3015(2)	10541(2)	29(1)
C(18)	7780(2)	3134(2)	9976(2)	25(1)
Cl(3)	9829(1)	1574(1)	3740(1)	25(1)
Cl(4)	7487(1)	1786(1)	7757(1)	31(1)
O(3)	873(2)	1470(1)	1883(1)	36(1)
O(4)	7711(2)	4703(1)	7813(1)	30(1)

Table A2.17. Selected bond lengths [Å] and angles [°] for [Ni(Htpt)Cl(H_2O)$_2$]Cl_2.2H_2O, **2.10 (C)**.

Ni-N(1)	2.0126(16)
Ni-O(2)	2.0768(16)
Ni-O(1)	2.0837(15)
Ni-N(4)	2.1566(17)
Ni-N(5)	2.1777(17)
Ni-Cl(1)	2.3206(5)
N(1)-Ni-O(2)	92.46(6)
N(1)-Ni-O(1)	91.14(6)
O(2)-Ni-O(1)	171.43(7)
N(1)-Ni-N(4)	76.57(6)
O(2)-Ni-N(4)	84.85(7)
O(1)-Ni-N(4)	88.43(6)
N(1)-Ni-N(5)	76.03(6)
O(2)-Ni-N(5)	95.17(7)
O(1)-Ni-N(5)	93.25(6)
N(4)-Ni-N(5)	152.57(6)
N(1)-Ni-Cl(1)	175.90(5)
O(2)-Ni-Cl(1)	89.42(5)
O(1)-Ni-Cl(1)	87.50(4)
N(4)-Ni-Cl(1)	107.25(5)
N(5)-Ni-Cl(1)	100.17(5)

Symmetry transformations used to generate equivalent atoms:

Table A2.18. Bond lengths [Å] and angles [°] for $[Ni(Htpt)Cl(H_2O)_2]Cl_2.2H_2O$, **2.10 (C)**.

Ni-N(1)	2.0126(16)
Ni-O(2)	2.0768(16)
Ni-O(1)	2.0837(15)
Ni-N(4)	2.1566(17)
Ni-N(5)	2.1777(17)
Ni-Cl(1)	2.3206(5)
O(1)-H(1A)	0.831(18)
O(1)-H(1B)	0.824(18)
O(2)-H(2A)	0.813(18)
O(2)-H(2B)	0.820(18)
N(1)-C(3)	1.332(3)
N(1)-C(1)	1.339(2)
N(2)-C(1)	1.333(3)
N(2)-C(2)	1.342(3)
N(3)-C(3)	1.335(3)
N(3)-C(2)	1.345(3)
N(4)-C(8)	1.341(3)
N(4)-C(4)	1.363(3)
N(5)-C(13)	1.343(3)
N(5)-C(9)	1.360(3)
N(6)-C(18)	1.338(3)
N(6)-C(14)	1.358(3)
N(6)-H(6X)	0.87(3)
C(1)-C(9)	1.487(3)
C(2)-C(14)	1.486(3)
C(3)-C(4)	1.480(3)
C(4)-C(5)	1.386(3)
C(5)-C(6)	1.391(3)
C(5)-H(5)	0.9500
C(6)-C(7)	1.392(3)
C(6)-H(6)	0.9500
C(7)-C(8)	1.393(3)
C(7)-H(7)	0.9500
C(8)-H(8)	0.9500
C(9)-C(10)	1.391(3)
C(10)-C(11)	1.391(3)

C(10)-H(10)	0.9500
C(11)-C(12)	1.386(3)
C(11)-H(11)	0.9500
C(12)-C(13)	1.399(3)
C(12)-H(12)	0.9500
C(13)-H(13)	0.9500
C(14)-C(15)	1.382(3)
C(15)-C(16)	1.400(3)
C(15)-H(15)	0.9500
C(16)-C(17)	1.389(3)
C(16)-H(16)	0.9500
C(17)-C(18)	1.388(3)
C(17)-H(17)	0.9500
C(18)-H(18)	0.9500
O(3)-H(3A)	0.811(19)
O(3)-H(3B)	0.812(19)
O(4)-H(4A)	0.830(18)
O(4)-H(4B)	0.808(19)

N(1)-Ni-O(2)	92.46(6)
N(1)-Ni-O(1)	91.14(6)
O(2)-Ni-O(1)	171.43(7)
N(1)-Ni-N(4)	76.57(6)
O(2)-Ni-N(4)	84.85(7)
O(1)-Ni-N(4)	88.43(6)
N(1)-Ni-N(5)	76.03(6)
O(2)-Ni-N(5)	95.17(7)
O(1)-Ni-N(5)	93.25(6)
N(4)-Ni-N(5)	152.57(6)
N(1)-Ni-Cl(1)	175.90(5)
O(2)-Ni-Cl(1)	89.42(5)
O(1)-Ni-Cl(1)	87.50(4)
N(4)-Ni-Cl(1)	107.25(5)
N(5)-Ni-Cl(1)	100.17(5)
Ni-O(1)-H(1A)	117.5(18)
Ni-O(1)-H(1B)	110.9(18)
H(1A)-O(1)-H(1B)	103(2)
Ni-O(2)-H(2A)	121.1(19)

Ni-O(2)-H(2B)	120.5(19)
H(2A)-O(2)-H(2B)	109(2)
C(3)-N(1)-C(1)	118.11(17)
C(3)-N(1)-Ni	120.46(13)
C(1)-N(1)-Ni	121.39(13)
C(1)-N(2)-C(2)	113.84(17)
C(3)-N(3)-C(2)	113.75(17)
C(8)-N(4)-C(4)	117.55(17)
C(8)-N(4)-Ni	127.89(14)
C(4)-N(4)-Ni	114.51(12)
C(13)-N(5)-C(9)	117.68(18)
C(13)-N(5)-Ni	127.74(14)
C(9)-N(5)-Ni	114.58(13)
C(18)-N(6)-C(14)	122.78(19)
C(18)-N(6)-H(6X)	117.8(17)
C(14)-N(6)-H(6X)	119.4(17)
N(2)-C(1)-N(1)	123.43(18)
N(2)-C(1)-C(9)	122.73(17)
N(1)-C(1)-C(9)	113.83(17)
N(2)-C(2)-N(3)	127.34(18)
N(2)-C(2)-C(14)	117.03(17)
N(3)-C(2)-C(14)	115.63(17)
N(1)-C(3)-N(3)	123.51(17)
N(1)-C(3)-C(4)	114.47(17)
N(3)-C(3)-C(4)	122.02(17)
N(4)-C(4)-C(5)	123.70(18)
N(4)-C(4)-C(3)	113.94(17)
C(5)-C(4)-C(3)	122.35(18)
C(4)-C(5)-C(6)	118.0(2)
C(4)-C(5)-H(5)	121.0
C(6)-C(5)-H(5)	121.0
C(5)-C(6)-C(7)	118.98(19)
C(5)-C(6)-H(6)	120.5
C(7)-C(6)-H(6)	120.5
C(6)-C(7)-C(8)	119.50(19)
C(6)-C(7)-H(7)	120.3
C(8)-C(7)-H(7)	120.3
N(4)-C(8)-C(7)	122.3(2)

N(4)-C(8)-H(8)	118.9
C(7)-C(8)-H(8)	118.9
N(5)-C(9)-C(10)	123.54(19)
N(5)-C(9)-C(1)	114.14(17)
C(10)-C(9)-C(1)	122.31(18)
C(11)-C(10)-C(9)	117.84(19)
C(11)-C(10)-H(10)	121.1
C(9)-C(10)-H(10)	121.1
C(12)-C(11)-C(10)	119.38(19)
C(12)-C(11)-H(11)	120.3
C(10)-C(11)-H(11)	120.3
C(11)-C(12)-C(13)	119.3(2)
C(11)-C(12)-H(12)	120.3
C(13)-C(12)-H(12)	120.3
N(5)-C(13)-C(12)	122.2(2)
N(5)-C(13)-H(13)	118.9
C(12)-C(13)-H(13)	118.9
N(6)-C(14)-C(15)	119.45(18)
N(6)-C(14)-C(2)	116.83(18)
C(15)-C(14)-C(2)	123.72(19)
C(14)-C(15)-C(16)	119.0(2)
C(14)-C(15)-H(15)	120.5
C(16)-C(15)-H(15)	120.5
C(17)-C(16)-C(15)	119.8(2)
C(17)-C(16)-H(16)	120.1
C(15)-C(16)-H(16)	120.1
C(18)-C(17)-C(16)	119.3(2)
C(18)-C(17)-H(17)	120.4
C(16)-C(17)-H(17)	120.4
N(6)-C(18)-C(17)	119.6(2)
N(6)-C(18)-H(18)	120.2
C(17)-C(18)-H(18)	120.2
H(3A)-O(3)-H(3B)	108(2)
H(4A)-O(4)-H(4B)	106(2)

Symmetry transformations used to generate equivalent atoms:

Table A2.19. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $[Ni(Htpt)Cl(H_2O)_2]Cl_2.2H_2O$, **2.10 (C)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	15(1)	13(1)	16(1)	0(1)	1(1)	2(1)
Cl(1)	28(1)	19(1)	21(1)	1(1)	1(1)	9(1)
O(1)	20(1)	19(1)	22(1)	1(1)	6(1)	1(1)
O(2)	44(1)	18(1)	23(1)	0(1)	13(1)	3(1)
N(1)	13(1)	16(1)	17(1)	1(1)	2(1)	0(1)
N(2)	16(1)	17(1)	17(1)	0(1)	3(1)	1(1)
N(3)	15(1)	15(1)	19(1)	-1(1)	4(1)	0(1)
N(4)	15(1)	19(1)	16(1)	-1(1)	3(1)	2(1)
N(5)	20(1)	17(1)	18(1)	1(1)	2(1)	-2(1)
N(6)	22(1)	18(1)	18(1)	1(1)	3(1)	4(1)
C(1)	14(1)	18(1)	14(1)	1(1)	3(1)	1(1)
C(2)	17(1)	18(1)	14(1)	1(1)	5(1)	3(1)
C(3)	16(1)	16(1)	14(1)	0(1)	5(1)	-1(1)
C(4)	14(1)	18(1)	16(1)	-1(1)	4(1)	-1(1)
C(5)	19(1)	21(1)	20(1)	-1(1)	5(1)	-2(1)
C(6)	16(1)	30(1)	23(1)	-3(1)	3(1)	-7(1)
C(7)	12(1)	37(1)	21(1)	1(1)	1(1)	1(1)
C(8)	15(1)	25(1)	19(1)	1(1)	3(1)	5(1)
C(9)	19(1)	18(1)	15(1)	0(1)	4(1)	-2(1)
C(10)	15(1)	24(1)	18(1)	1(1)	4(1)	0(1)
C(11)	20(1)	31(1)	21(1)	-2(1)	4(1)	-9(1)
C(12)	30(1)	21(1)	26(1)	-3(1)	5(1)	-10(1)
C(13)	26(1)	18(1)	23(1)	1(1)	4(1)	-2(1)
C(14)	22(1)	18(1)	15(1)	0(1)	6(1)	2(1)
C(15)	19(1)	22(1)	20(1)	2(1)	5(1)	4(1)
C(16)	23(1)	31(1)	27(1)	5(1)	5(1)	10(1)
C(17)	35(1)	22(1)	29(1)	6(1)	10(1)	13(1)
C(18)	35(1)	17(1)	22(1)	-2(1)	8(1)	3(1)
Cl(3)	30(1)	19(1)	27(1)	1(1)	10(1)	5(1)
Cl(4)	35(1)	20(1)	42(1)	-5(1)	16(1)	-7(1)
O(3)	36(1)	43(1)	31(1)	-4(1)	14(1)	-1(1)
O(4)	39(1)	19(1)	34(1)	3(1)	15(1)	5(1)

Table A2.20. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $[Ni(Htpt)Cl(H_2O)_2]Cl_2.2H_2O$, **2.10 (C)**.

	x	y	z	U(eq)
H(1A)	6324(19)	7917(13)	7360(20)	31
H(1B)	6280(20)	8806(13)	7360(20)	31
H(2A)	5000(20)	8024(14)	10530(20)	42
H(2B)	5030(20)	8939(14)	10510(20)	42
H(6X)	6640(20)	4037(17)	9481(19)	26(6)
H(5)	4172	5164	8358	24
H(6)	2263	5441	7440	28
H(7)	1641	6969	7104	29
H(8)	2921	8172	7621	25
H(10)	9577	7701	10954	36(7)
H(11)	10286	9212	11282	26(6)
H(12)	9083	10457	10639	22(6)
H(13)	7216	10182	9661	28
H(15)	9531	5189	11002	25
H(16)	10322	3707	11340	33
H(17)	9219	2416	10655	34
H(18)	7320	2617	9708	30
H(3A)	1290(20)	1913(18)	2020(20)	54
H(3B)	490(20)	1470(20)	2280(20)	54
H(4A)	7089(18)	4469(18)	7520(20)	44
H(4B)	7680(20)	5225(14)	7600(20)	44

Table A2.21. Hydrogen bonds for $[Ni(Htpt)Cl(H_2O)_2]Cl_2.2H_2O$, **2.10 (C)** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(4)-H(4B)...Cl(4)#1	0.808(19)	2.318(19)	3.1174(17)	170(3)
O(4)-H(4A)...O(3)#2	0.830(18)	2.029(19)	2.829(3)	162(3)
O(3)-H(3B)...Cl(3)#3	0.812(19)	2.34(2)	3.1331(19)	166(3)
O(3)-H(3A)...Cl(4)#4	0.811(19)	2.43(2)	3.233(2)	169(3)
N(6)-H(6X)...Cl(3)#5	0.87(3)	2.35(3)	3.1443(19)	152(2)
O(2)-H(2B)...Cl(1)#6	0.820(18)	2.290(19)	3.1064(16)	174(2)
O(2)-H(2A)...Cl(3)#1	0.813(18)	2.307(18)	3.1025(17)	166(3)
O(1)-H(1B)...O(4)#1	0.824(18)	1.876(19)	2.691(2)	170(2)
O(1)-H(1A)...Cl(4)#1	0.831(18)	2.251(18)	3.0803(16)	175(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x+3/2,y+1/2,-z+3/2 #2 x+1/2,-y+1/2,z+1/2

#3 x-1,y,z #4 x-1/2,-y+1/2,z-1/2 #5 x-1/2,-y+1/2,z+1/2

#6 -x+1,-y+2,-z+2

Table A2.22. Crystal data and structure refinement for $[Ni(tpt)(H_2O)_3](NO_3)_2$, **2.12 (D)**.

Identification code	**2.12 (D)**	
Empirical formula	C18 H18 N8 Ni O9	
Formula weight	549.11	
Temperature	164(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.4053(8) Å	α= 63.4740(10)°.
	b = 13.3116(14) Å	β= 77.8480(10)°.
	c = 13.8265(15) Å	γ = 84.5990(10)°.
Volume	1192.2(2) $Å^3$	
Z	2	
Density (calculated)	1.530 Mg/m^3	
Absorption coefficient	0.879 mm^{-1}	
F(000)	564	
Crystal size	0.52 x 0.20 x 0.04 mm^3	
Theta range for data collection	1.68 to 26.50°.	
Index ranges	-9<=h<=4, -16<=k<=16, -17<=l<=17	
Reflections collected	14726	
Independent reflections	4726 [R(int) = 0.0235]	
Completeness to theta = 26.50°	95.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9657 and 0.6579	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4726 / 0 / 327	
Goodness-of-fit on F^2	1.114	
Final R indices [I>2sigma(I)]	R1 = 0.0468, wR2 = 0.1208	
R indices (all data)	R1 = 0.0599, wR2 = 0.1256	
Largest diff. peak and hole	1.476 and -0.949 e.$Å^{-3}$	

Table A2.23. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[Ni(tpt)(H_2O)_3](NO_3)_2$, **2.12 (D)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni	9106(1)	3405(1)	3091(1)	22(1)
N(1)	9421(3)	3145(2)	1752(2)	20(1)
N(2)	8710(3)	3656(2)	8(2)	23(1)
N(3)	10426(3)	1963(2)	890(2)	25(1)
N(4)	10790(3)	1921(2)	3494(2)	24(1)
N(5)	7572(3)	4773(2)	2034(2)	23(1)
N(6)	9134(4)	3262(2)	-1803(2)	32(1)
C(1)	8620(4)	3821(2)	901(2)	21(1)
C(2)	9634(4)	2714(2)	42(2)	22(1)
C(3)	10284(4)	2220(2)	1729(2)	20(1)
C(4)	11061(4)	1503(2)	2740(2)	22(1)
C(5)	11984(4)	497(2)	2885(3)	27(1)
C(6)	12693(4)	-101(3)	3857(3)	31(1)
C(7)	12461(4)	334(3)	4616(3)	31(1)
C(8)	11512(4)	1338(3)	4413(2)	28(1)
C(9)	7583(4)	4777(2)	1047(2)	22(1)
C(10)	6683(4)	5591(2)	240(2)	25(1)
C(11)	5734(4)	6450(3)	465(3)	30(1)
C(12)	5706(5)	6449(3)	1466(3)	31(1)
C(13)	6637(4)	5606(3)	2232(3)	27(1)
C(14)	9785(4)	2464(3)	-921(2)	27(1)
C(15)	10552(6)	1461(3)	-900(3)	40(1)
C(16)	10643(7)	1248(3)	-1807(3)	57(1)
C(17)	9952(7)	2049(3)	-2705(3)	58(1)
C(18)	9226(6)	3047(3)	-2677(3)	46(1)
O(1)	6708(3)	2411(2)	3807(2)	28(1)
O(2)	11484(3)	4394(2)	2487(2)	29(1)
O(3)	8714(4)	3540(2)	4537(2)	38(1)
N'	3253(8)	4014(3)	4869(3)	79(2)
O(1')	3648(4)	3688(2)	4134(2)	47(1)
O(2')	1830(8)	4540(4)	4958(4)	134(3)
O(3')	4543(10)	3733(5)	5499(4)	153(3)

N"	6218(4)	1255(3)	2071(2)	37(1)
O(1")	6986(4)	539(3)	2823(2)	54(1)
O(2")	5589(4)	2149(2)	2115(2)	45(1)
O(3")	6102(4)	1098(2)	1256(2)	47(1)

Table A2.24. Selected bond lengths [Å] and angles [°] for $[Ni(tpt)(H_2O)_3](NO_3)_2$, **2.12 (D)**.

Ni-N(1)	1.993(2)
Ni-O(3)	2.045(2)
Ni-O(2)	2.090(2)
Ni-O(1)	2.101(2)
Ni-N(4)	2.148(2)
Ni-N(5)	2.162(2)
N(1)-Ni-O(3)	175.40(9)
N(1)-Ni-O(2)	92.96(9)
O(3)-Ni-O(2)	90.64(9)
N(1)-Ni-O(1)	91.08(9)
O(3)-Ni-O(1)	85.29(9)
O(2)-Ni-O(1)	175.90(8)
N(1)-Ni-N(4)	77.22(9)
O(3)-Ni-N(4)	99.93(10)
O(2)-Ni-N(4)	89.88(9)
O(1)-Ni-N(4)	90.33(9)
N(1)-Ni-N(5)	76.90(9)
O(3)-Ni-N(5)	105.91(10)
O(2)-Ni-N(5)	91.27(9)
O(1)-Ni-N(5)	90.32(9)
N(4)-Ni-N(5)	154.12(10)

Symmetry transformations used to generate equivalent atoms:

Table A2.25. Bond lengths [Å] and angles [°] for $[Ni(tpt)(H_2O)_3](NO_3)_2$, **2.12 (D)**.

Ni-N(1)	1.993(2)
Ni-O(3)	2.045(2)
Ni-O(2)	2.090(2)
Ni-O(1)	2.101(2)
Ni-N(4)	2.148(2)
Ni-N(5)	2.162(2)
N(1)-C(1)	1.339(4)
N(1)-C(3)	1.343(4)
N(2)-C(1)	1.335(4)
N(2)-C(2)	1.357(4)
N(3)-C(3)	1.331(4)
N(3)-C(2)	1.360(4)
N(4)-C(8)	1.353(4)
N(4)-C(4)	1.359(4)
N(5)-C(13)	1.353(4)
N(5)-C(9)	1.361(4)
N(6)-C(18)	1.349(4)
N(6)-C(14)	1.357(4)
C(1)-C(9)	1.493(4)
C(2)-C(14)	1.491(4)
C(3)-C(4)	1.497(4)
C(4)-C(5)	1.394(4)
C(5)-C(6)	1.406(4)
C(5)-H(5)	0.9500
C(6)-C(7)	1.385(5)
C(6)-H(6)	0.9500
C(7)-C(8)	1.389(5)
C(7)-H(7)	0.9500
C(8)-H(8)	0.9500
C(9)-C(10)	1.400(4)
C(10)-C(11)	1.404(4)
C(10)-H(10)	0.9500
C(11)-C(12)	1.379(5)
C(11)-H(11)	0.9500
C(12)-C(13)	1.399(4)
C(12)-H(12)	0.9500

C(13)-H(13)	0.9500
C(14)-C(15)	1.392(5)
C(15)-C(16)	1.390(5)
C(15)-H(15)	0.9500
C(16)-C(17)	1.387(6)
C(16)-H(16)	0.9500
C(17)-C(18)	1.399(6)
C(17)-H(17)	0.9500
C(18)-H(18)	0.9500
O(1)-H(1A)	0.8199
O(1)-H(1B)	0.8818
O(2)-H(2A)	0.8120
O(2)-H(2B)	0.8959
O(3)-H(3A)	0.8616
O(3)-H(3B)	0.8387
N'-O(2')	1.225(7)
N'-O(1')	1.246(4)
N'-O(3')	1.342(8)
N''-O(1'')	1.254(4)
N''-O(3'')	1.254(4)
N''-O(2'')	1.259(4)

N(1)-Ni-O(3)	175.40(9)
N(1)-Ni-O(2)	92.96(9)
O(3)-Ni-O(2)	90.64(9)
N(1)-Ni-O(1)	91.08(9)
O(3)-Ni-O(1)	85.29(9)
O(2)-Ni-O(1)	175.90(8)
N(1)-Ni-N(4)	77.22(9)
O(3)-Ni-N(4)	99.93(10)
O(2)-Ni-N(4)	89.88(9)
O(1)-Ni-N(4)	90.33(9)
N(1)-Ni-N(5)	76.90(9)
O(3)-Ni-N(5)	105.91(10)
O(2)-Ni-N(5)	91.27(9)
O(1)-Ni-N(5)	90.32(9)
N(4)-Ni-N(5)	154.12(10)
C(1)-N(1)-C(3)	118.0(2)

C(1)-N(1)-Ni	121.10(19)
C(3)-N(1)-Ni	120.52(19)
C(1)-N(2)-C(2)	114.5(2)
C(3)-N(3)-C(2)	114.9(2)
C(8)-N(4)-C(4)	117.4(3)
C(8)-N(4)-Ni	128.2(2)
C(4)-N(4)-Ni	114.23(19)
C(13)-N(5)-C(9)	117.4(3)
C(13)-N(5)-Ni	128.4(2)
C(9)-N(5)-Ni	114.18(18)
C(18)-N(6)-C(14)	117.5(3)
N(2)-C(1)-N(1)	123.7(3)
N(2)-C(1)-C(9)	122.6(3)
N(1)-C(1)-C(9)	113.7(2)
N(2)-C(2)-N(3)	125.6(3)
N(2)-C(2)-C(14)	117.8(3)
N(3)-C(2)-C(14)	116.6(3)
N(3)-C(3)-N(1)	123.3(3)
N(3)-C(3)-C(4)	123.2(2)
N(1)-C(3)-C(4)	113.6(2)
N(4)-C(4)-C(5)	123.4(3)
N(4)-C(4)-C(3)	114.0(2)
C(5)-C(4)-C(3)	122.6(3)
C(4)-C(5)-C(6)	117.9(3)
C(4)-C(5)-H(5)	121.0
C(6)-C(5)-H(5)	121.0
C(7)-C(6)-C(5)	118.9(3)
C(7)-C(6)-H(6)	120.5
C(5)-C(6)-H(6)	120.5
C(6)-C(7)-C(8)	119.6(3)
C(6)-C(7)-H(7)	120.2
C(8)-C(7)-H(7)	120.2
N(4)-C(8)-C(7)	122.7(3)
N(4)-C(8)-H(8)	118.7
C(7)-C(8)-H(8)	118.7
N(5)-C(9)-C(10)	123.4(3)
N(5)-C(9)-C(1)	114.0(2)
C(10)-C(9)-C(1)	122.6(3)

C(9)-C(10)-C(11)	117.9(3)
C(9)-C(10)-H(10)	121.0
C(11)-C(10)-H(10)	121.0
C(12)-C(11)-C(10)	119.1(3)
C(12)-C(11)-H(11)	120.5
C(10)-C(11)-H(11)	120.5
C(11)-C(12)-C(13)	119.7(3)
C(11)-C(12)-H(12)	120.1
C(13)-C(12)-H(12)	120.1
N(5)-C(13)-C(12)	122.4(3)
N(5)-C(13)-H(13)	118.8
C(12)-C(13)-H(13)	118.8
N(6)-C(14)-C(15)	122.6(3)
N(6)-C(14)-C(2)	116.4(3)
C(15)-C(14)-C(2)	121.0(3)
C(16)-C(15)-C(14)	119.4(4)
C(16)-C(15)-H(15)	120.3
C(14)-C(15)-H(15)	120.3
C(17)-C(16)-C(15)	118.5(4)
C(17)-C(16)-H(16)	120.8
C(15)-C(16)-H(16)	120.8
C(16)-C(17)-C(18)	119.1(3)
C(16)-C(17)-H(17)	120.5
C(18)-C(17)-H(17)	120.5
N(6)-C(18)-C(17)	122.9(4)
N(6)-C(18)-H(18)	118.5
C(17)-C(18)-H(18)	118.5
Ni-O(1)-H(1A)	109.4
Ni-O(1)-H(1B)	113.7
H(1A)-O(1)-H(1B)	96.8
Ni-O(2)-H(2A)	109.4
Ni-O(2)-H(2B)	123.9
H(2A)-O(2)-H(2B)	107.1
Ni-O(3)-H(3A)	127.2
Ni-O(3)-H(3B)	109.4
H(3A)-O(3)-H(3B)	102.0
O(2')-N'-O(1')	120.9(5)
O(2')-N'-O(3')	126.6(5)

O(1')-N'-O(3')	112.6(6)
O(1")-N"-O(3")	120.4(3)
O(1")-N"-O(2")	120.4(3)
O(3")-N"-O(2")	119.2(3)

Symmetry transformations used to generate equivalent atoms:

Table A2.26. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $[Ni(tpt)(H_2O)_3](NO_3)_2$, **2.12 (D)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	27(1)	20(1)	19(1)	-9(1)	-5(1)	2(1)
N(1)	21(1)	19(1)	21(1)	-7(1)	-5(1)	1(1)
N(2)	27(1)	21(1)	21(1)	-9(1)	-3(1)	-4(1)
N(3)	24(1)	22(1)	28(1)	-13(1)	-3(1)	-2(1)
N(4)	23(1)	23(1)	26(1)	-10(1)	-5(1)	-1(1)
N(5)	26(1)	21(1)	23(1)	-11(1)	-5(1)	1(1)
N(6)	44(2)	29(1)	23(1)	-13(1)	-3(1)	-8(1)
C(1)	21(2)	19(1)	22(1)	-7(1)	-4(1)	-3(1)
C(2)	21(2)	22(1)	23(2)	-10(1)	-1(1)	-5(1)
C(3)	19(2)	19(1)	23(1)	-10(1)	-1(1)	-2(1)
C(4)	21(2)	20(1)	23(2)	-8(1)	-2(1)	-3(1)
C(5)	25(2)	22(2)	32(2)	-11(1)	-5(1)	1(1)
C(6)	26(2)	22(2)	37(2)	-5(1)	-8(1)	2(1)
C(7)	26(2)	30(2)	28(2)	-3(1)	-8(1)	-1(1)
C(8)	28(2)	29(2)	22(2)	-7(1)	-6(1)	-4(1)
C(9)	20(2)	22(1)	24(2)	-11(1)	-2(1)	-3(1)
C(10)	22(2)	24(2)	27(2)	-8(1)	-7(1)	-2(1)
C(11)	26(2)	24(2)	38(2)	-10(1)	-10(1)	3(1)
C(12)	29(2)	24(2)	41(2)	-17(1)	-5(1)	5(1)
C(13)	27(2)	26(2)	30(2)	-16(1)	-3(1)	2(1)
C(14)	33(2)	25(2)	22(2)	-11(1)	2(1)	-7(1)
C(15)	59(2)	28(2)	34(2)	-18(2)	-6(2)	3(2)
C(16)	92(4)	40(2)	47(2)	-32(2)	0(2)	0(2)
C(17)	106(4)	45(2)	29(2)	-25(2)	2(2)	-10(2)
C(18)	72(3)	43(2)	26(2)	-20(2)	-3(2)	-11(2)
O(1)	28(1)	26(1)	28(1)	-10(1)	-3(1)	0(1)
O(2)	36(1)	23(1)	28(1)	-8(1)	-10(1)	-3(1)
O(3)	56(2)	35(1)	28(1)	-19(1)	-3(1)	-6(1)
N'	158(5)	54(2)	27(2)	-23(2)	11(2)	-50(3)
O(1')	59(2)	50(2)	45(2)	-28(1)	-24(1)	16(1)
O(2')	167(5)	99(3)	146(4)	-106(3)	106(4)	-74(3)
O(3')	253(7)	169(5)	61(3)	-51(3)	-34(4)	-91(5)
N''	33(2)	49(2)	26(2)	-13(1)	-2(1)	-13(1)

O(1")	46(2)	74(2)	31(1)	-13(1)	-12(1)	10(1)
O(2")	52(2)	40(2)	42(2)	-18(1)	-1(1)	-16(1)
O(3")	53(2)	56(2)	39(2)	-24(1)	-15(1)	1(1)

Table A2.27. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $[Ni(tpt)(H_2O)_3](NO_3)_2$, **2.12 (D)**.

	x	y	z	U(eq)
H(5)	12129	225	2345	32
H(6)	13321	-793	3990	37
H(7)	12948	-51	5272	37
H(8)	11364	1627	4941	33
H(10)	6712	5564	-438	29
H(11)	5121	7025	-65	36
H(12)	5057	7019	1635	37
H(13)	6611	5618	2916	32
H(15)	11008	926	-272	48
H(16)	11168	571	-1811	69
H(17)	9971	1922	-3331	70
H(18)	8778	3597	-3302	55
H(1A)	6422	2246	3354	42
H(1B)	5695	2789	3899	42
H(2A)	12077	4210	2972	44
H(2B)	11477	5146	2147	44
H(3A)	9190	4032	4655	57
H(3B)	7582	3607	4753	57

Table A2.28. Hydrogen bonds for $[Ni(tpt)(H_2O)_3](NO_3)_2$, **2.12 (D)** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1A)...O(2")	0.82	2.00	2.807(3)	169.1
O(1)-H(1A)...O(1")	0.82	2.66	3.304(4)	137.0
O(1)-H(1A)...N"	0.82	2.67	3.468(4)	163.6
O(1)-H(1B)...O(1')	0.88	1.91	2.786(3)	174.4
O(2)-H(2A)...O(1')#1	0.81	2.01	2.824(3)	176.2
O(2)-H(2B)...N(6)#2	0.90	1.98	2.849(3)	162.2
O(3)-H(3A)...O(2')#3	0.86	2.22	2.909(4)	136.6
O(3)-H(3A)...O(2')#1	0.86	2.30	3.045(6)	144.4
O(3)-H(3B)...O(3')	0.84	2.29	3.130(8)	174.8

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z #2 -x+2,-y+1,-z #3 -x+1,-y+1,-z+1

Table A2.29. Crystal data and structure refinement for $[Ni(tpt)_2](ClO_4)_2$, **2.13 (E)**.

Identification code	**2.13 (E)**	
Empirical formula	C36 H24 Cl2 N12 Ni O8	
Formula weight	882.28	
Temperature	163(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P c a 21	
Unit cell dimensions	a = 15.886(3) Å	α= 90°.
	b = 11.519(2) Å	β= 90°.
	c = 21.636(4) Å	γ = 90°.
Volume	3959.3(13) $Å^3$	
Z	4	
Density (calculated)	1.480 Mg/m^3	
Absorption coefficient	0.692 mm^{-1}	
F(000)	1800	
Crystal size	0.75 x 0.19 x 0.09 mm^3	
Theta range for data collection	1.77 to 26.82°.	
Index ranges	-9<=h<=19, -14<=k<=14, -26<=l<=27	
Reflections collected	40287	
Independent reflections	7833 [R(int) = 0.0609]	
Completeness to theta = 26.82°	95.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9403 and 0.6249	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7833 / 1 / 532	
Goodness-of-fit on F^2	0.965	
Final R indices [I>2sigma(I)]	R1 = 0.0378, wR2 = 0.0718	
R indices (all data)	R1 = 0.0680, wR2 = 0.0816	
Absolute structure parameter	0.008(11)	
Largest diff. peak and hole	0.300 and -0.325 e.$Å^{-3}$	

Table A2.30. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[Ni(tpt)_2](ClO_4)_2$, **2.13 (E)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni	4100(1)	119(1)	4259(1)	25(1)
N(1)	3942(2)	-1129(2)	3592(1)	26(1)
N(2)	3191(2)	-1811(2)	2688(1)	29(1)
N(3)	4242(2)	-3052(2)	3201(1)	30(1)
N(4)	4994(2)	-1277(2)	4532(1)	27(1)
N(5)	3154(2)	900(2)	3629(1)	27(1)
N(6)	3023(2)	-3527(2)	1760(1)	41(1)
C(1)	3368(2)	-977(3)	3117(1)	24(1)
C(2)	3633(2)	-2834(3)	2746(1)	29(1)
C(3)	4361(2)	-2171(3)	3613(1)	25(1)
C(4)	4972(2)	-2271(3)	4158(1)	27(1)
C(5)	5456(2)	-3282(3)	4276(2)	33(1)
C(6)	6007(2)	-3273(3)	4797(2)	38(1)
C(7)	6048(2)	-2276(3)	5171(2)	39(1)
C(8)	5534(2)	-1297(3)	5030(2)	36(1)
C(9)	2927(2)	187(3)	3129(1)	25(1)
C(10)	2327(2)	536(3)	2675(1)	30(1)
C(11)	1950(2)	1663(3)	2722(2)	38(1)
C(12)	2180(2)	2387(3)	3228(2)	42(1)
C(13)	2780(2)	1974(3)	3674(2)	35(1)
C(14)	3426(2)	-3828(3)	2304(2)	32(1)
C(15)	3623(2)	-4995(3)	2467(2)	39(1)
C(16)	3380(3)	-5913(3)	2057(2)	46(1)
C(17)	2957(2)	-5615(3)	1503(2)	47(1)
C(18)	2796(2)	-4423(3)	1378(2)	44(1)
N(1')	4196(2)	1384(2)	4925(1)	25(1)
N(2')	3692(2)	2225(2)	5882(1)	28(1)
N(3')	4881(2)	3109(2)	5313(1)	30(1)
N(4')	5134(2)	1236(2)	3921(1)	27(1)
N(5')	3153(2)	-410(2)	4956(1)	28(1)
N(6')	4103(2)	3882(3)	6846(1)	41(1)
C(1')	3676(2)	1385(3)	5437(1)	26(1)

C(2')	4305(2)	3069(3)	5793(1)	30(1)
C(3')	4780(2)	2255(3)	4883(1)	25(1)
C(4')	5326(2)	2179(2)	4302(2)	26(1)
C(5')	5958(2)	3012(3)	4156(2)	31(1)
C(6')	6418(2)	2864(3)	3595(2)	33(1)
C(7')	6233(2)	1917(3)	3207(2)	34(1)
C(8')	5590(2)	1114(3)	3383(1)	31(1)
C(9')	3090(2)	345(3)	5462(1)	26(1)
C(10')	2552(2)	118(3)	5975(2)	31(1)
C(11')	2076(2)	-933(3)	5980(2)	37(1)
C(12')	2131(2)	-1688(3)	5466(2)	35(1)
C(13')	2663(2)	-1388(3)	4963(2)	34(1)
C(14')	4335(2)	4097(3)	6243(1)	31(1)
C(15')	4570(2)	5212(3)	6013(2)	43(1)
C(16')	4560(3)	6178(3)	6422(2)	52(1)
C(17')	4345(3)	5981(4)	7044(2)	55(1)
C(18')	4129(3)	4837(3)	7239(2)	53(1)
Cl(1)	3225(1)	4909(1)	4420(1)	37(1)
O(11)	3004(2)	4158(2)	4952(1)	48(1)
O(12)	3370(2)	6103(2)	4625(2)	72(1)
O(13)	2525(2)	4882(2)	3963(1)	55(1)
O(14)	3996(2)	4443(2)	4127(1)	52(1)
Cl(2)	4951(1)	163(1)	1763(1)	39(1)
O(21)	4905(2)	-696(3)	2269(1)	68(1)
O(22)	5748(2)	807(3)	1808(1)	57(1)
O(23)	4240(2)	983(3)	1806(2)	82(1)
O(24)	4906(2)	-463(3)	1169(1)	59(1)

Table A2.31. Selected bond lengths [Å] and angles [°] for $[Ni(tpt)_2](ClO_4)_2$, **2.13 (E)**.

Ni-N(1)	2.053(3)
Ni-N(1')	2.054(2)
Ni-N(4')	2.211(3)
Ni-N(5')	2.216(3)
Ni-N(5)	2.220(3)
Ni-N(4)	2.225(3)
N(1)-Ni-N(1')	177.21(10)
N(1)-Ni-N(4')	105.41(10)
N(1')-Ni-N(4')	76.35(9)
N(1)-Ni-N(5')	101.70(10)
N(1')-Ni-N(5')	76.62(10)
N(4')-Ni-N(5')	152.84(9)
N(1)-Ni-N(5)	76.67(10)
N(1')-Ni-N(5)	101.14(10)
N(4')-Ni-N(5)	93.68(10)
N(5')-Ni-N(5)	93.96(9)
N(1)-Ni-N(4)	76.03(10)
N(1')-Ni-N(4)	106.20(10)
N(4')-Ni-N(4)	91.95(9)
N(5')-Ni-N(4)	93.11(10)
N(5)-Ni-N(4)	152.65(10)

Symmetry transformations used to generate equivalent atoms:

Table A2.32. Bond lengths [Å] and angles [°] for $[Ni(tpt)_2](ClO_4)_2$, **2.13 (E)**.

Ni-N(1)	2.053(3)
Ni-N(1')	2.054(2)
Ni-N(4')	2.211(3)
Ni-N(5')	2.216(3)
Ni-N(5)	2.220(3)
Ni-N(4)	2.225(3)
N(1)-C(3)	1.373(4)
N(1)-C(1)	1.385(4)
N(2)-C(1)	1.365(4)
N(2)-C(2)	1.377(4)
N(3)-C(3)	1.365(4)
N(3)-C(2)	1.403(4)
N(4)-C(8)	1.377(4)
N(4)-C(4)	1.403(4)
N(5)-C(13)	1.375(4)
N(5)-C(9)	1.406(4)
N(6)-C(18)	1.370(4)
N(6)-C(14)	1.384(4)
C(1)-C(9)	1.513(4)
C(2)-C(14)	1.527(4)
C(3)-C(4)	1.531(4)
C(4)-C(5)	1.419(4)
C(5)-C(6)	1.427(4)
C(5)-H(5)	0.9500
C(6)-C(7)	1.405(5)
C(6)-H(6)	0.9500
C(7)-C(8)	1.426(5)
C(7)-H(7)	0.9500
C(8)-H(8)	0.9500
C(9)-C(10)	1.426(5)
C(10)-C(11)	1.433(5)
C(10)-H(10)	0.9500
C(11)-C(12)	1.425(5)
C(11)-H(11)	0.9500
C(12)-C(13)	1.437(5)
C(12)-H(12)	0.9500

C(13)-H(13)	0.9500
C(14)-C(15)	1.424(5)
C(15)-C(16)	1.433(5)
C(15)-H(15)	0.9500
C(16)-C(17)	1.418(5)
C(16)-H(16)	0.9500
C(17)-C(18)	1.423(5)
C(17)-H(17)	0.9500
C(18)-H(18)	0.9500
N(1')-C(3')	1.370(4)
N(1')-C(1')	1.383(4)
N(2')-C(1')	1.365(4)
N(2')-C(2')	1.390(4)
N(3')-C(3')	1.364(4)
N(3')-C(2')	1.384(4)
N(4')-C(8')	1.378(4)
N(4')-C(4')	1.398(4)
N(5')-C(13')	1.369(4)
N(5')-C(9')	1.402(4)
N(6')-C(14')	1.379(4)
N(6')-C(18')	1.391(4)
C(1')-C(9')	1.517(4)
C(2')-C(14')	1.534(4)
C(3')-C(4')	1.529(4)
C(4')-C(5')	1.424(4)
C(5')-C(6')	1.426(5)
C(5')-H(5')	0.9500
C(6')-C(7')	1.408(4)
C(6')-H(6')	0.9500
C(7')-C(8')	1.430(5)
C(7')-H(7')	0.9500
C(8')-H(8')	0.9500
C(9')-C(10')	1.425(5)
C(10')-C(11')	1.427(5)
C(10')-H(10')	0.9500
C(11')-C(12')	1.414(5)
C(11')-H(11')	0.9500
C(12')-C(13')	1.421(5)

C(12')-H(12')	0.9500
C(13')-H(13')	0.9500
C(14')-C(15')	1.427(5)
C(15')-C(16')	1.422(5)
C(15')-H(15')	0.9500
C(16')-C(17')	1.408(5)
C(16')-H(16')	0.9500
C(17')-C(18')	1.426(6)
C(17')-H(17')	0.9500
C(18')-H(18')	0.9500
Cl(1)-O(12)	1.463(3)
Cl(1)-O(14)	1.481(3)
Cl(1)-O(11)	1.482(3)
Cl(1)-O(13)	1.489(3)
Cl(2)-O(22)	1.471(3)
Cl(2)-O(21)	1.476(3)
Cl(2)-O(23)	1.475(3)
Cl(2)-O(24)	1.476(3)

N(1)-Ni-N(1')	177.21(10)
N(1)-Ni-N(4')	105.41(10)
N(1')-Ni-N(4')	76.35(9)
N(1)-Ni-N(5')	101.70(10)
N(1')-Ni-N(5')	76.62(10)
N(4')-Ni-N(5')	152.84(9)
N(1)-Ni-N(5)	76.67(10)
N(1')-Ni-N(5)	101.14(10)
N(4')-Ni-N(5)	93.68(10)
N(5')-Ni-N(5)	93.96(9)
N(1)-Ni-N(4)	76.03(10)
N(1')-Ni-N(4)	106.20(10)
N(4')-Ni-N(4)	91.95(9)
N(5')-Ni-N(4)	93.11(10)
N(5)-Ni-N(4)	152.65(10)
C(3)-N(1)-C(1)	117.0(3)
C(3)-N(1)-Ni	122.0(2)
C(1)-N(1)-Ni	120.9(2)
C(1)-N(2)-C(2)	115.9(3)

C(3)-N(3)-C(2)	114.9(3)
C(8)-N(4)-C(4)	117.0(3)
C(8)-N(4)-Ni	128.1(2)
C(4)-N(4)-Ni	114.88(19)
C(13)-N(5)-C(9)	117.9(3)
C(13)-N(5)-Ni	127.9(2)
C(9)-N(5)-Ni	114.16(19)
C(18)-N(6)-C(14)	116.4(3)
N(2)-C(1)-N(1)	123.4(3)
N(2)-C(1)-C(9)	122.7(3)
N(1)-C(1)-C(9)	113.9(3)
N(2)-C(2)-N(3)	124.6(3)
N(2)-C(2)-C(14)	118.4(3)
N(3)-C(2)-C(14)	117.0(3)
N(3)-C(3)-N(1)	124.2(3)
N(3)-C(3)-C(4)	122.3(3)
N(1)-C(3)-C(4)	113.5(2)
N(4)-C(4)-C(5)	123.5(3)
N(4)-C(4)-C(3)	113.5(2)
C(5)-C(4)-C(3)	123.0(3)
C(4)-C(5)-C(6)	118.0(3)
C(4)-C(5)-H(5)	121.0
C(6)-C(5)-H(5)	121.0
C(7)-C(6)-C(5)	119.3(3)
C(7)-C(6)-H(6)	120.4
C(5)-C(6)-H(6)	120.4
C(6)-C(7)-C(8)	119.8(3)
C(6)-C(7)-H(7)	120.1
C(8)-C(7)-H(7)	120.1
N(4)-C(8)-C(7)	122.5(3)
N(4)-C(8)-H(8)	118.7
C(7)-C(8)-H(8)	118.7
N(5)-C(9)-C(10)	122.5(3)
N(5)-C(9)-C(1)	114.3(3)
C(10)-C(9)-C(1)	123.2(3)
C(9)-C(10)-C(11)	119.0(3)
C(9)-C(10)-H(10)	120.5
C(11)-C(10)-H(10)	120.5

C(12)-C(11)-C(10)	118.5(3)
C(12)-C(11)-H(11)	120.7
C(10)-C(11)-H(11)	120.7
C(11)-C(12)-C(13)	119.5(3)
C(11)-C(12)-H(12)	120.2
C(13)-C(12)-H(12)	120.2
N(5)-C(13)-C(12)	122.5(3)
N(5)-C(13)-H(13)	118.7
C(12)-C(13)-H(13)	118.7
N(6)-C(14)-C(15)	123.3(3)
N(6)-C(14)-C(2)	116.3(3)
C(15)-C(14)-C(2)	120.4(3)
C(16)-C(15)-C(14)	119.0(3)
C(16)-C(15)-H(15)	120.5
C(14)-C(15)-H(15)	120.5
C(17)-C(16)-C(15)	118.2(4)
C(17)-C(16)-H(16)	120.9
C(15)-C(16)-H(16)	120.9
C(16)-C(17)-C(18)	118.6(3)
C(16)-C(17)-H(17)	120.7
C(18)-C(17)-H(17)	120.7
N(6)-C(18)-C(17)	124.5(3)
N(6)-C(18)-H(18)	117.8
C(17)-C(18)-H(18)	117.8
C(3')-N(1')-C(1')	117.2(3)
C(3')-N(1')-Ni	121.61(19)
C(1')-N(1')-Ni	121.2(2)
C(1')-N(2')-C(2')	114.3(3)
C(3')-N(3')-C(2')	114.2(3)
C(8')-N(4')-C(4')	117.6(3)
C(8')-N(4')-Ni	127.6(2)
C(4')-N(4')-Ni	114.79(19)
C(13')-N(5')-C(9')	117.5(3)
C(13')-N(5')-Ni	128.3(2)
C(9')-N(5')-Ni	114.1(2)
C(14')-N(6')-C(18')	115.4(3)
N(2')-C(1')-N(1')	123.7(3)
N(2')-C(1')-C(9')	123.1(3)

N(1')-C(1')-C(9')	113.2(3)
N(3')-C(2')-N(2')	126.2(3)
N(3')-C(2')-C(14')	115.5(3)
N(2')-C(2')-C(14')	118.3(3)
N(3')-C(3')-N(1')	124.3(3)
N(3')-C(3')-C(4')	122.4(3)
N(1')-C(3')-C(4')	113.3(2)
N(4')-C(4')-C(5')	123.1(3)
N(4')-C(4')-C(3')	113.9(2)
C(5')-C(4')-C(3')	122.9(3)
C(4')-C(5')-C(6')	118.0(3)
C(4')-C(5')-H(5')	121.0
C(6')-C(5')-H(5')	121.0
C(7')-C(6')-C(5')	119.6(3)
C(7')-C(6')-H(6')	120.2
C(5')-C(6')-H(6')	120.2
C(6')-C(7')-C(8')	119.4(3)
C(6')-C(7')-H(7')	120.3
C(8')-C(7')-H(7')	120.3
N(4')-C(8')-C(7')	122.4(3)
N(4')-C(8')-H(8')	118.8
C(7')-C(8')-H(8')	118.8
N(5')-C(9')-C(10')	122.4(3)
N(5')-C(9')-C(1')	114.7(3)
C(10')-C(9')-C(1')	122.8(3)
C(9')-C(10')-C(11')	118.7(3)
C(9')-C(10')-H(10')	120.7
C(11')-C(10')-H(10')	120.7
C(12')-C(11')-C(10')	118.9(3)
C(12')-C(11')-H(11')	120.5
C(10')-C(11')-H(11')	120.5
C(11')-C(12')-C(13')	119.2(3)
C(11')-C(12')-H(12')	120.4
C(13')-C(12')-H(12')	120.4
N(5')-C(13')-C(12')	123.2(3)
N(5')-C(13')-H(13')	118.4
C(12')-C(13')-H(13')	118.4
N(6')-C(14')-C(15')	124.2(3)

N(6')-C(14')-C(2')	117.0(3)
C(15')-C(14')-C(2')	118.7(3)
C(14')-C(15')-C(16')	118.9(3)
C(14')-C(15')-H(15')	120.5
C(16')-C(15')-H(15')	120.5
C(17')-C(16')-C(15')	118.2(4)
C(17')-C(16')-H(16')	120.9
C(15')-C(16')-H(16')	120.9
C(16')-C(17')-C(18')	119.4(3)
C(16')-C(17')-H(17')	120.3
C(18')-C(17')-H(17')	120.3
N(6')-C(18')-C(17')	123.8(3)
N(6')-C(18')-H(18')	118.1
C(17')-C(18')-H(18')	118.1
O(12)-Cl(1)-O(14)	109.86(17)
O(12)-Cl(1)-O(11)	110.61(18)
O(14)-Cl(1)-O(11)	108.50(15)
O(12)-Cl(1)-O(13)	109.76(17)
O(14)-Cl(1)-O(13)	108.96(16)
O(11)-Cl(1)-O(13)	109.12(15)
O(22)-Cl(2)-O(21)	109.29(17)
O(22)-Cl(2)-O(23)	109.36(19)
O(21)-Cl(2)-O(23)	110.2(2)
O(22)-Cl(2)-O(24)	110.22(18)
O(21)-Cl(2)-O(24)	108.44(17)
O(23)-Cl(2)-O(24)	109.33(17)

Symmetry transformations used to generate equivalent atoms:

Table A2.33. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $[Ni(tpt)_2](ClO_4)_2$, **2.13 (E)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	27(1)	25(1)	24(1)	-4(1)	-2(1)	0(1)
N(1)	23(2)	26(2)	28(1)	1(1)	1(1)	2(1)
N(2)	27(2)	32(2)	27(1)	-5(1)	2(1)	1(1)
N(3)	32(2)	28(2)	28(1)	-5(1)	3(1)	3(1)
N(4)	29(2)	26(2)	28(1)	-1(1)	-2(1)	1(1)
N(5)	31(2)	26(2)	24(1)	-1(1)	0(1)	1(1)
N(6)	48(2)	37(2)	36(2)	-10(1)	-8(2)	-1(2)
C(1)	26(2)	26(2)	21(2)	-2(1)	1(1)	-2(1)
C(2)	28(2)	34(2)	25(2)	-5(2)	2(1)	-3(2)
C(3)	25(2)	26(2)	23(2)	-1(1)	4(1)	0(1)
C(4)	21(2)	29(2)	30(2)	0(1)	2(1)	-2(1)
C(5)	33(2)	26(2)	38(2)	-4(2)	2(2)	2(1)
C(6)	29(2)	35(2)	49(2)	8(2)	-6(2)	8(2)
C(7)	35(2)	40(2)	42(2)	4(2)	-17(2)	-2(2)
C(8)	39(2)	35(2)	33(2)	-2(2)	-11(2)	-2(2)
C(9)	28(2)	22(2)	26(2)	0(1)	2(1)	-6(1)
C(10)	33(2)	32(2)	24(2)	1(1)	-4(2)	-1(2)
C(11)	43(2)	32(2)	38(2)	10(2)	-6(2)	3(2)
C(12)	46(2)	33(2)	48(2)	-2(2)	-5(2)	11(2)
C(13)	41(2)	32(2)	33(2)	-5(2)	-3(2)	5(2)
C(14)	33(2)	28(2)	34(2)	-5(2)	3(2)	-4(2)
C(15)	46(2)	36(2)	36(2)	-8(2)	-1(2)	-1(2)
C(16)	55(3)	26(2)	57(3)	-13(2)	9(2)	-6(2)
C(17)	47(2)	46(2)	47(2)	-22(2)	3(2)	-5(2)
C(18)	43(2)	46(2)	41(2)	-13(2)	-6(2)	-2(2)
N(1')	29(2)	25(2)	22(1)	-1(1)	0(1)	-1(1)
N(2')	34(2)	25(2)	25(1)	-5(1)	-3(1)	0(1)
N(3')	35(2)	27(2)	29(2)	-3(1)	-3(1)	1(1)
N(4')	29(2)	25(2)	28(2)	-4(1)	0(1)	-1(1)
N(5')	31(2)	26(2)	27(2)	-2(1)	0(1)	-4(1)
N(6')	47(2)	42(2)	35(2)	-11(1)	1(1)	-5(2)
C(1')	29(2)	24(2)	25(2)	2(1)	-2(1)	5(1)
C(2')	34(2)	28(2)	28(2)	0(1)	-3(2)	2(2)

C(3')	26(2)	24(2)	24(2)	1(1)	-4(1)	3(1)
C(4')	27(2)	24(2)	26(2)	-4(2)	-1(2)	2(1)
C(5')	32(2)	29(2)	33(2)	-5(1)	-5(2)	-4(2)
C(6')	31(2)	35(2)	35(2)	2(2)	5(2)	-4(2)
C(7')	29(2)	41(2)	31(2)	-2(2)	3(2)	3(2)
C(8')	33(2)	30(2)	28(2)	-5(1)	1(2)	3(2)
C(9')	28(2)	24(2)	24(2)	1(1)	2(1)	3(2)
C(10')	38(2)	28(2)	26(2)	-3(1)	0(1)	2(2)
C(11')	35(2)	43(2)	32(2)	5(2)	5(2)	1(2)
C(12')	38(2)	29(2)	39(2)	4(2)	0(2)	-8(2)
C(13')	33(2)	33(2)	35(2)	-5(2)	-3(2)	-1(2)
C(14')	33(2)	33(2)	27(2)	-8(1)	0(1)	-3(2)
C(15')	53(3)	35(2)	40(2)	-8(2)	7(2)	-6(2)
C(16')	62(3)	34(2)	58(3)	-14(2)	10(2)	-10(2)
C(17')	66(3)	50(3)	50(2)	-27(2)	12(2)	-10(2)
C(18')	62(3)	60(3)	37(2)	-18(2)	7(2)	-9(2)
Cl(1)	42(1)	28(1)	41(1)	0(1)	0(1)	3(1)
O(11)	39(2)	60(2)	45(2)	13(1)	-3(1)	-6(1)
O(12)	84(2)	34(2)	97(2)	-25(2)	13(2)	-9(2)
O(13)	54(2)	56(2)	56(2)	9(1)	-18(1)	11(1)
O(14)	46(2)	52(2)	58(2)	6(1)	8(1)	16(1)
Cl(2)	32(1)	54(1)	32(1)	-5(1)	3(1)	-1(1)
O(21)	92(2)	71(2)	40(2)	11(2)	4(2)	-37(2)
O(22)	39(2)	66(2)	66(2)	15(2)	2(1)	-16(1)
O(23)	43(2)	88(2)	113(3)	-49(2)	-3(2)	20(2)
O(24)	64(2)	79(2)	33(1)	-13(1)	0(1)	19(2)

Table A2.34. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $[Ni(tpt)_2](ClO_4)_2$, **2.13 (E)**.

	x	y	z	U(eq)
H(5)	5415	-3945	4017	39
H(6)	6342	-3933	4891	46
H(7)	6417	-2255	5516	47
H(8)	5565	-633	5289	43
H(10)	2180	28	2346	36
H(11)	1555	1921	2422	45
H(12)	1940	3138	3270	51
H(13)	2924	2461	4012	42
H(15)	3912	-5162	2841	47
H(16)	3499	-6700	2155	55
H(17)	2785	-6199	1220	56
H(18)	2511	-4235	1005	52
H(5')	6070	3647	4424	37
H(6')	6846	3401	3485	40
H(7')	6533	1813	2831	40
H(8')	5474	473	3120	37
H(10')	2511	655	6307	37
H(11')	1729	-1122	6323	44
H(12')	1815	-2388	5457	42
H(13')	2680	-1890	4615	40
H(15')	4731	5307	5593	51
H(16')	4695	6935	6279	62
H(17')	4344	6604	7331	66
H(18')	3994	4720	7663	63

Table A2.35. Crystal data and structure refinement for $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15 (F)**.

Identification code	**2.15 (F)**	
Empirical formula	C22 H26 N10 Ni2 O15	
Formula weight	787.95	
Temperature	108(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.3769(3) Å	α= 73.018(2)°.
	b = 14.0212(4) Å	β= 84.192(2)°.
	c = 15.1672(5) Å	γ = 89.706(2)°.
Volume	1492.19(9) $Å^3$	
Z	2	
Density (calculated)	1.754 Mg/m^3	
Absorption coefficient	1.353 mm^{-1}	
F(000)	808	
Crystal size	0.31 x 0.14 x 0.04 mm^3	
Theta range for data collection	2.36 to 26.62°.	
Index ranges	-9<=h<=9, -16<=k<=17, -19<=l<=18	
Reflections collected	13017	
Independent reflections	5974 [R(int) = 0.0285]	
Completeness to theta = 26.62°	95.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9479 and 0.6792	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5974 / 0 / 457	
Goodness-of-fit on F^2	1.031	
Final R indices [I>2sigma(I)]	R1 = 0.0359, wR2 = 0.0834	
R indices (all data)	R1 = 0.0546, wR2 = 0.0917	
Largest diff. peak and hole	0.618 and -0.464 e.$Å^{-3}$	

Table A2.36. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15 (F)**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni(1)	1810(1)	-1418(1)	2495(1)	27(1)
Ni(2)	5358(1)	2670(1)	1982(1)	28(1)
C(1)	3140(3)	568(2)	2304(2)	24(1)
C(2)	3539(3)	1159(2)	3507(2)	26(1)
C(3)	2507(3)	-392(2)	3793(2)	26(1)
C(4)	1861(4)	-1366(2)	4397(2)	28(1)
C(5)	1642(4)	-1574(2)	5336(2)	35(1)
C(6)	1031(4)	-2514(3)	5843(2)	43(1)
C(7)	672(5)	-3190(3)	5392(2)	47(1)
C(8)	900(4)	-2923(2)	4446(2)	41(1)
C(9)	3017(3)	585(2)	1336(2)	25(1)
C(10)	3287(4)	1415(2)	597(2)	33(1)
C(11)	3055(4)	1333(3)	-268(2)	41(1)
C(12)	2570(4)	443(3)	-361(2)	43(1)
C(13)	2287(4)	-357(2)	410(2)	37(1)
C(14)	3994(4)	1982(2)	3863(2)	29(1)
C(15)	3659(4)	1899(2)	4786(2)	37(1)
C(16)	4013(5)	2704(3)	5080(2)	44(1)
C(17)	4671(5)	3565(3)	4451(2)	45(1)
C(18)	5006(4)	3582(2)	3545(2)	40(1)
N(1)	2575(3)	-289(2)	2900(1)	24(1)
N(2)	3713(3)	1322(2)	2583(1)	25(1)
N(3)	2956(3)	324(2)	4130(2)	30(1)
N(4)	1482(3)	-2023(2)	3944(2)	30(1)
N(5)	2496(3)	-298(2)	1248(2)	29(1)
N(6)	4684(3)	2807(2)	3249(2)	31(1)
N(7)	-1885(3)	-611(2)	2926(2)	32(1)
O(71)	-909(3)	-1039(2)	2427(1)	35(1)
O(72)	-1241(3)	-303(2)	3502(1)	41(1)
O(73)	-3517(3)	-520(2)	2811(2)	52(1)
N(8)	1808(3)	3466(2)	1522(2)	41(1)
O(81)	3469(3)	3408(2)	1217(1)	37(1)

O(82)	675(3)	3644(2)	979(2)	69(1)
O(83)	1402(4)	3343(4)	2319(2)	110(2)
N(9)	7944(3)	1716(2)	1536(2)	41(1)
O(91)	7669(3)	1858(2)	2318(2)	41(1)
O(92)	6788(3)	2101(2)	984(2)	44(1)
O(93)	9196(3)	1244(2)	1331(2)	66(1)
N(10)	3063(4)	3887(2)	6746(2)	50(1)
O(101)	2471(4)	3086(2)	7247(2)	84(1)
O(102)	2045(5)	4515(3)	6416(3)	109(1)
O(103)	4734(4)	3986(2)	6588(2)	68(1)
O(1)	4417(3)	-1920(2)	2391(2)	45(1)
O(2)	925(3)	-2550(2)	2083(2)	50(1)
C(21)	1854(6)	-3228(4)	1686(4)	83(2)
C(22)	919(9)	-3679(6)	1186(6)	158(4)
O(3)	7075(3)	3890(2)	1583(2)	40(1)
C(31)	6879(5)	4764(3)	838(2)	51(1)
C(32)	5913(8)	5535(4)	1139(3)	88(2)

Table A2.37. Selected bond lengths [Å] and angles [°] for $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15 (F)**.

Ni(1)-N(1)	1.962(2)
Ni(1)-O(2)	2.001(2)
Ni(1)-O(1)	2.051(2)
Ni(1)-O(71)	2.0771(19)
Ni(1)-N(5)	2.093(2)
Ni(1)-N(4)	2.101(2)
Ni(2)-O(81)	1.993(2)
Ni(2)-N(6)	1.999(2)
Ni(2)-O(3)	2.040(2)
Ni(2)-O(91)	2.069(2)
Ni(2)-O(92)	2.101(2)
Ni(2)-N(2)	2.161(2)
N(1)-Ni(1)-O(2)	177.67(10)
N(1)-Ni(1)-O(1)	92.84(10)
O(2)-Ni(1)-O(1)	89.49(10)
N(1)-Ni(1)-O(71)	96.39(8)
O(2)-Ni(1)-O(71)	81.29(9)
O(1)-Ni(1)-O(71)	169.81(9)
N(1)-Ni(1)-N(5)	76.62(9)
O(2)-Ni(1)-N(5)	103.38(10)
O(1)-Ni(1)-N(5)	88.41(10)
O(71)-Ni(1)-N(5)	89.51(8)
N(1)-Ni(1)-N(4)	78.21(9)
O(2)-Ni(1)-N(4)	101.77(10)
O(1)-Ni(1)-N(4)	92.70(10)
O(71)-Ni(1)-N(4)	93.43(8)
N(5)-Ni(1)-N(4)	154.83(9)
O(81)-Ni(2)-N(6)	105.16(9)
O(81)-Ni(2)-O(3)	91.77(9)
N(6)-Ni(2)-O(3)	93.71(9)
O(81)-Ni(2)-O(91)	159.45(9)
N(6)-Ni(2)-O(91)	95.35(9)
O(3)-Ni(2)-O(91)	85.53(9)
O(81)-Ni(2)-O(92)	98.59(9)

N(6)-Ni(2)-O(92)	155.85(10)
O(3)-Ni(2)-O(92)	89.78(9)
O(91)-Ni(2)-O(92)	61.08(9)
O(81)-Ni(2)-N(2)	95.13(8)
N(6)-Ni(2)-N(2)	80.62(8)
O(3)-Ni(2)-N(2)	172.01(9)
O(91)-Ni(2)-N(2)	89.38(8)
O(92)-Ni(2)-N(2)	93.15(9)

Symmetry transformations used to generate equivalent atoms:

Table A2.38. Bond lengths [Å] and angles [°] for $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15 (F)**.

Ni(1)-N(1)	1.962(2)
Ni(1)-O(2)	2.001(2)
Ni(1)-O(1)	2.051(2)
Ni(1)-O(71)	2.0771(19)
Ni(1)-N(5)	2.093(2)
Ni(1)-N(4)	2.101(2)
Ni(2)-O(81)	1.993(2)
Ni(2)-N(6)	1.999(2)
Ni(2)-O(3)	2.040(2)
Ni(2)-O(91)	2.069(2)
Ni(2)-O(92)	2.101(2)
Ni(2)-N(2)	2.161(2)
C(1)-N(1)	1.316(3)
C(1)-N(2)	1.334(3)
C(1)-C(9)	1.472(3)
C(2)-N(3)	1.314(3)
C(2)-N(2)	1.345(3)
C(2)-C(14)	1.463(4)
C(3)-N(3)	1.310(3)
C(3)-N(1)	1.315(3)
C(3)-C(4)	1.455(4)
C(4)-N(4)	1.346(3)
C(4)-C(5)	1.361(4)
C(5)-C(6)	1.370(4)
C(5)-H(5)	0.9500
C(6)-C(7)	1.362(5)
C(6)-H(6)	0.9500
C(7)-C(8)	1.365(5)
C(7)-H(7)	0.9500
C(8)-N(4)	1.317(4)
C(8)-H(8)	0.9500
C(9)-N(5)	1.345(3)
C(9)-C(10)	1.358(4)
C(10)-C(11)	1.378(4)
C(10)-H(10)	0.9500
C(11)-C(12)	1.351(5)

C(11)-H(11)	0.9500
C(12)-C(13)	1.362(4)
C(12)-H(12)	0.9500
C(13)-N(5)	1.323(3)
C(13)-H(13)	0.9500
C(14)-N(6)	1.321(4)
C(14)-C(15)	1.367(4)
C(15)-C(16)	1.365(4)
C(15)-H(15)	0.9500
C(16)-C(17)	1.357(5)
C(16)-H(16)	0.9500
C(17)-C(18)	1.365(4)
C(17)-H(17)	0.9500
C(18)-N(6)	1.322(4)
C(18)-H(18)	0.9500
N(7)-O(72)	1.218(3)
N(7)-O(73)	1.234(3)
N(7)-O(71)	1.266(3)
N(8)-O(83)	1.177(4)
N(8)-O(82)	1.207(3)
N(8)-O(81)	1.276(3)
N(9)-O(93)	1.201(3)
N(9)-O(91)	1.256(3)
N(9)-O(92)	1.263(3)
N(10)-O(102)	1.184(4)
N(10)-O(101)	1.213(4)
N(10)-O(103)	1.232(4)
O(1)-H(1A)	0.8400
O(1)-H(1B)	0.70(4)
O(2)-C(21)	1.409(4)
O(2)-H(2)	0.81(4)
C(21)-C(22)	1.356(7)
C(21)-H(21A)	0.9900
C(21)-H(21B)	0.9900
C(22)-H(22A)	0.9800
C(22)-H(22B)	0.9800
C(22)-H(22C)	0.9800
O(3)-C(31)	1.423(4)

O(3)-H(3)	0.74(4)
C(31)-C(32)	1.449(5)
C(31)-H(31A)	0.9900
C(31)-H(31B)	0.9900
C(32)-H(32A)	0.9800
C(32)-H(32B)	0.9800
C(32)-H(32C)	0.9800

N(1)-Ni(1)-O(2)	177.67(10)
N(1)-Ni(1)-O(1)	92.84(10)
O(2)-Ni(1)-O(1)	89.49(10)
N(1)-Ni(1)-O(71)	96.39(8)
O(2)-Ni(1)-O(71)	81.29(9)
O(1)-Ni(1)-O(71)	169.81(9)
N(1)-Ni(1)-N(5)	76.62(9)
O(2)-Ni(1)-N(5)	103.38(10)
O(1)-Ni(1)-N(5)	88.41(10)
O(71)-Ni(1)-N(5)	89.51(8)
N(1)-Ni(1)-N(4)	78.21(9)
O(2)-Ni(1)-N(4)	101.77(10)
O(1)-Ni(1)-N(4)	92.70(10)
O(71)-Ni(1)-N(4)	93.43(8)
N(5)-Ni(1)-N(4)	154.83(9)
O(81)-Ni(2)-N(6)	105.16(9)
O(81)-Ni(2)-O(3)	91.77(9)
N(6)-Ni(2)-O(3)	93.71(9)
O(81)-Ni(2)-O(91)	159.45(9)
N(6)-Ni(2)-O(91)	95.35(9)
O(3)-Ni(2)-O(91)	85.53(9)
O(81)-Ni(2)-O(92)	98.59(9)
N(6)-Ni(2)-O(92)	155.85(10)
O(3)-Ni(2)-O(92)	89.78(9)
O(91)-Ni(2)-O(92)	61.08(9)
O(81)-Ni(2)-N(2)	95.13(8)
N(6)-Ni(2)-N(2)	80.62(8)
O(3)-Ni(2)-N(2)	172.01(9)
O(91)-Ni(2)-N(2)	89.38(8)
O(92)-Ni(2)-N(2)	93.15(9)

N(1)-C(1)-N(2)	121.5(2)
N(1)-C(1)-C(9)	112.6(2)
N(2)-C(1)-C(9)	125.9(2)
N(3)-C(2)-N(2)	125.8(2)
N(3)-C(2)-C(14)	116.1(2)
N(2)-C(2)-C(14)	118.1(2)
N(3)-C(3)-N(1)	123.4(2)
N(3)-C(3)-C(4)	121.4(2)
N(1)-C(3)-C(4)	115.1(2)
N(4)-C(4)-C(5)	123.8(3)
N(4)-C(4)-C(3)	114.2(2)
C(5)-C(4)-C(3)	122.1(3)
C(4)-C(5)-C(6)	117.5(3)
C(4)-C(5)-H(5)	121.2
C(6)-C(5)-H(5)	121.2
C(7)-C(6)-C(5)	119.2(3)
C(7)-C(6)-H(6)	120.4
C(5)-C(6)-H(6)	120.4
C(6)-C(7)-C(8)	120.0(3)
C(6)-C(7)-H(7)	120.0
C(8)-C(7)-H(7)	120.0
N(4)-C(8)-C(7)	121.9(3)
N(4)-C(8)-H(8)	119.0
C(7)-C(8)-H(8)	119.0
N(5)-C(9)-C(10)	122.0(2)
N(5)-C(9)-C(1)	113.5(2)
C(10)-C(9)-C(1)	124.4(2)
C(9)-C(10)-C(11)	118.3(3)
C(9)-C(10)-H(10)	120.8
C(11)-C(10)-H(10)	120.8
C(12)-C(11)-C(10)	119.8(3)
C(12)-C(11)-H(11)	120.1
C(10)-C(11)-H(11)	120.1
C(11)-C(12)-C(13)	119.0(3)
C(11)-C(12)-H(12)	120.5
C(13)-C(12)-H(12)	120.5
N(5)-C(13)-C(12)	122.3(3)
N(5)-C(13)-H(13)	118.8

C(12)-C(13)-H(13)	118.8
N(6)-C(14)-C(15)	122.3(3)
N(6)-C(14)-C(2)	116.8(2)
C(15)-C(14)-C(2)	120.8(3)
C(16)-C(15)-C(14)	118.8(3)
C(16)-C(15)-H(15)	120.6
C(14)-C(15)-H(15)	120.6
C(17)-C(16)-C(15)	119.3(3)
C(17)-C(16)-H(16)	120.4
C(15)-C(16)-H(16)	120.4
C(16)-C(17)-C(18)	118.4(3)
C(16)-C(17)-H(17)	120.8
C(18)-C(17)-H(17)	120.8
N(6)-C(18)-C(17)	123.1(3)
N(6)-C(18)-H(18)	118.4
C(17)-C(18)-H(18)	118.4
C(3)-N(1)-C(1)	119.3(2)
C(3)-N(1)-Ni(1)	118.97(18)
C(1)-N(1)-Ni(1)	121.77(17)
C(1)-N(2)-C(2)	114.9(2)
C(1)-N(2)-Ni(2)	136.83(17)
C(2)-N(2)-Ni(2)	106.97(16)
C(3)-N(3)-C(2)	115.0(2)
C(8)-N(4)-C(4)	117.6(3)
C(8)-N(4)-Ni(1)	128.9(2)
C(4)-N(4)-Ni(1)	113.43(18)
C(13)-N(5)-C(9)	118.5(2)
C(13)-N(5)-Ni(1)	125.8(2)
C(9)-N(5)-Ni(1)	115.33(17)
C(14)-N(6)-C(18)	118.0(2)
C(14)-N(6)-Ni(2)	114.16(18)
C(18)-N(6)-Ni(2)	127.5(2)
O(72)-N(7)-O(73)	121.1(2)
O(72)-N(7)-O(71)	121.3(2)
O(73)-N(7)-O(71)	117.6(2)
N(7)-O(71)-Ni(1)	128.31(16)
O(83)-N(8)-O(82)	121.2(3)
O(83)-N(8)-O(81)	120.2(3)

O(82)-N(8)-O(81)	118.6(3)
N(8)-O(81)-Ni(2)	124.41(17)
O(93)-N(9)-O(91)	122.8(3)
O(93)-N(9)-O(92)	122.7(3)
O(91)-N(9)-O(92)	114.6(2)
N(9)-O(91)-Ni(2)	93.03(16)
N(9)-O(92)-Ni(2)	91.34(17)
O(102)-N(10)-O(101)	119.9(4)
O(102)-N(10)-O(103)	123.5(4)
O(101)-N(10)-O(103)	116.6(3)
Ni(1)-O(1)-H(1A)	109.5
Ni(1)-O(1)-H(1B)	121(3)
H(1A)-O(1)-H(1B)	112.2
C(21)-O(2)-Ni(1)	132.0(2)
C(21)-O(2)-H(2)	111(2)
Ni(1)-O(2)-H(2)	114(2)
C(22)-C(21)-O(2)	118.0(5)
C(22)-C(21)-H(21A)	107.8
O(2)-C(21)-H(21A)	107.8
C(22)-C(21)-H(21B)	107.8
O(2)-C(21)-H(21B)	107.8
H(21A)-C(21)-H(21B)	107.1
C(21)-C(22)-H(22A)	109.5
C(21)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(21)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
C(31)-O(3)-Ni(2)	125.5(2)
C(31)-O(3)-H(3)	106(3)
Ni(2)-O(3)-H(3)	113(3)
O(3)-C(31)-C(32)	112.4(3)
O(3)-C(31)-H(31A)	109.1
C(32)-C(31)-H(31A)	109.1
O(3)-C(31)-H(31B)	109.1
C(32)-C(31)-H(31B)	109.1
H(31A)-C(31)-H(31B)	107.8
C(31)-C(32)-H(32A)	109.5

C(31)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(31)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table A2.39. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15 (F)**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1)	26(1)	31(1)	27(1)	-12(1)	-4(1)	0(1)
Ni(2)	25(1)	32(1)	25(1)	-7(1)	0(1)	0(1)
C(1)	16(1)	33(2)	23(1)	-9(1)	-1(1)	2(1)
C(2)	26(1)	31(2)	23(1)	-8(1)	-1(1)	-1(1)
C(3)	21(1)	34(2)	22(1)	-7(1)	-3(1)	0(1)
C(4)	26(1)	31(2)	25(1)	-6(1)	-3(1)	0(1)
C(5)	34(2)	43(2)	26(2)	-7(1)	-3(1)	-1(1)
C(6)	43(2)	51(2)	28(2)	1(1)	-3(1)	-3(1)
C(7)	50(2)	37(2)	43(2)	7(2)	-5(2)	-7(1)
C(8)	45(2)	32(2)	45(2)	-7(1)	-13(1)	-3(1)
C(9)	20(1)	36(2)	22(1)	-11(1)	-2(1)	1(1)
C(10)	33(2)	39(2)	26(2)	-9(1)	-4(1)	-2(1)
C(11)	44(2)	53(2)	23(2)	-5(1)	-2(1)	-3(1)
C(12)	47(2)	62(2)	22(2)	-15(2)	-6(1)	-3(2)
C(13)	40(2)	47(2)	29(2)	-18(1)	-7(1)	0(1)
C(14)	30(1)	32(2)	26(1)	-11(1)	-1(1)	-1(1)
C(15)	45(2)	39(2)	26(2)	-13(1)	1(1)	-7(1)
C(16)	56(2)	51(2)	31(2)	-21(2)	1(1)	-6(2)
C(17)	59(2)	42(2)	43(2)	-25(2)	-1(2)	-5(2)
C(18)	50(2)	32(2)	38(2)	-12(1)	-1(1)	-7(1)
N(1)	22(1)	30(1)	20(1)	-8(1)	-3(1)	0(1)
N(2)	25(1)	30(1)	19(1)	-7(1)	-1(1)	-1(1)
N(3)	37(1)	33(1)	20(1)	-8(1)	-2(1)	-5(1)
N(4)	30(1)	29(1)	31(1)	-7(1)	-6(1)	0(1)
N(5)	26(1)	38(1)	25(1)	-14(1)	-4(1)	2(1)
N(6)	34(1)	31(1)	29(1)	-11(1)	-2(1)	-4(1)
N(7)	24(1)	34(1)	33(1)	-4(1)	-1(1)	-1(1)
O(71)	27(1)	44(1)	40(1)	-20(1)	-8(1)	4(1)
O(72)	36(1)	59(2)	34(1)	-22(1)	-7(1)	5(1)
O(73)	22(1)	67(2)	72(2)	-30(1)	-11(1)	5(1)
N(8)	30(1)	46(2)	42(2)	-3(1)	-1(1)	2(1)
O(81)	23(1)	46(1)	35(1)	-3(1)	0(1)	2(1)

O(82)	34(1)	101(2)	64(2)	-6(2)	-13(1)	6(1)
O(83)	50(2)	222(4)	43(2)	-20(2)	9(1)	36(2)
N(9)	30(1)	41(2)	53(2)	-18(1)	3(1)	-2(1)
O(91)	39(1)	42(1)	42(1)	-11(1)	-10(1)	5(1)
O(92)	34(1)	63(2)	39(1)	-21(1)	-1(1)	2(1)
O(93)	41(2)	63(2)	98(2)	-35(2)	10(1)	14(1)
N(10)	58(2)	37(2)	62(2)	-21(2)	-13(2)	1(1)
O(101)	59(2)	42(2)	125(3)	5(2)	19(2)	3(1)
O(102)	97(3)	65(2)	145(4)	5(2)	-39(2)	27(2)
O(103)	56(2)	68(2)	83(2)	-28(2)	2(1)	-14(1)
O(1)	32(1)	51(2)	56(2)	-20(1)	-9(1)	10(1)
O(2)	40(1)	51(2)	73(2)	-41(1)	0(1)	-3(1)
C(21)	63(3)	84(3)	134(4)	-82(3)	-7(3)	8(2)
C(22)	124(6)	202(8)	214(8)	-176(7)	25(5)	-23(5)
O(3)	31(1)	36(1)	47(1)	-3(1)	0(1)	-2(1)
C(31)	54(2)	44(2)	47(2)	-3(2)	-4(2)	-4(2)
C(32)	128(5)	73(3)	69(3)	-29(3)	-15(3)	36(3)

Table A2.40. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15 (F)**.

	x	y	z	U(eq)
H(5)	1904	-1085	5630	42
H(6)	859	-2692	6500	52
H(7)	263	-3847	5735	57
H(8)	632	-3401	4141	49
H(10)	3627	2035	675	39
H(11)	3236	1901	-797	49
H(12)	2427	375	-956	51
H(13)	1928	-980	343	44
H(15)	3189	1291	5215	44
H(16)	3801	2663	5719	53
H(17)	4893	4141	4636	55
H(18)	5494	4179	3106	48
H(1A)	5151	-1497	2458	67
H(1B)	4630(60)	-2410(30)	2630(30)	55(15)
H(2)	-90(50)	-2750(30)	2300(20)	42(10)
H(21A)	2922	-2870	1277	100
H(21B)	2327	-3757	2192	100
H(22A)	-249	-3945	1538	237
H(22B)	1632	-4225	1061	237
H(22C)	697	-3192	599	237
H(3)	8040(60)	3760(30)	1530(30)	63(14)
H(31A)	6209	4588	371	61
H(31B)	8101	5027	536	61
H(32A)	4768	5255	1508	132
H(32B)	5649	6078	596	132
H(32C)	6669	5794	1517	132

Table A2.41. Hydrogen bonds for $[Ni_2(tpt)(EtOH)_2(NO_3)_3(H_2O)](NO_3)$, **2.15 (F)** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1A)...O(73)#1	0.84	1.92	2.745(3)	168.7
O(1)-H(1B)...O(103)#2	0.70(4)	2.25(4)	2.956(4)	179(5)
O(1)-H(1B)...O(101)#2	0.70(4)	2.34(4)	2.817(4)	127(4)
O(1)-H(1B)...N(10)#2	0.70(4)	2.69(4)	3.333(4)	154(4)
O(2)-H(2)...O(101)#3	0.81(4)	1.83(4)	2.633(4)	174(4)
O(3)-H(3)...O(82)#1	0.74(4)	2.06(4)	2.773(4)	163(4)

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z #2 -x+1,-y,-z+1 #3 -x,-y,-z+1

Table A2.42. Crystal data and structure refinement for [Ru(tpm)(bpy)Cl](PF_6), **3.10**.

Identification code	3.10
Empirical formula	C20 H18 Cl F6 N8 P Ru
Formula weight	651.91
Temperature	88(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, *p* 21/*n*
Unit cell dimensions	a = 10.8239(9) A alpha = 90 deg. b = 15.0829(13) A beta = 97.314(2) deg. c = 14.3141(12) A gamma = 90 deg.
Volume	2317.8(3) A^3
Z, Calculated density	4, 1.868 Mg/m^3
Absorption coefficient	0.938 mm^-1
F(000)	1296
Crystal size	0.74 x 0.30 x 0.05 mm
Theta range for data collection	1.97 to 26.40 deg.
Limiting indices	-13<=h<=13, -18<=k<=18, -17<=l<=17
Reflections collected / unique	18093 / 4720 [R(int) = 0.0338]
Completeness to theta = 26.40	99.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9546 and 0.5436
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4720 / 0 / 334
Goodness-of-fit on F^2	1.046
Final R indices [I>2sigma(I)]	R1 = 0.0274, wR2 = 0.0650
R indices (all data)	R1 = 0.0435, wR2 = 0.0720
Largest diff. peak and hole	0.654 and -0.617 e.A^-3

Table A2.43. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for [Ru(tpm)(bpy)Cl](PF_6), **3.10.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ru	7574(1)	3152(1)	5602(1)	12(1)
N(1)	9277(2)	3208(2)	6431(2)	15(1)
N(2)	10093(2)	2541(2)	6334(2)	16(1)
N(3)	7595(2)	1839(2)	6015(2)	15(1)
N(4)	8663(2)	1377(2)	5974(2)	16(1)
N(5)	8546(2)	2780(2)	4551(2)	15(1)
N(6)	9457(2)	2158(2)	4740(2)	16(1)
N(7)	7491(2)	4420(2)	5112(2)	15(1)
N(8)	5925(2)	3127(2)	4730(2)	16(1)
Cl	6566(1)	3567(1)	6945(1)	12(1)
C(1)	11142(3)	2625(2)	6953(2)	19(1)
C(2)	11011(3)	3375(2)	7453(2)	21(1)
C(3)	9841(3)	3715(2)	7111(2)	19(1)
C(4)	8577(3)	547(2)	6316(2)	21(1)
C(5)	7415(3)	458(2)	6562(2)	22(1)
C(6)	6832(3)	1273(2)	6370(2)	19(1)
C(7)	10058(3)	2005(2)	3981(2)	20(1)
C(8)	9511(3)	2537(2)	3279(2)	21(1)
C(9)	8588(3)	3007(2)	3660(2)	19(1)
C(10)	9722(3)	1823(2)	5693(2)	17(1)
C(11)	8359(3)	5052(2)	5317(2)	20(1)
C(12)	8237(3)	5897(2)	4971(2)	25(1)
C(13)	7180(3)	6121(2)	4381(2)	25(1)
C(14)	6291(3)	5483(2)	4142(2)	22(1)
C(15)	6462(3)	4637(2)	4511(2)	17(1)
C(16)	5586(3)	3904(2)	4294(2)	17(1)
C(17)	4492(3)	3975(2)	3680(2)	22(1)
C(18)	3735(3)	3242(2)	3513(2)	23(1)
C(19)	4080(3)	2457(2)	3959(2)	23(1)
C(20)	5184(3)	2423(2)	4562(2)	19(1)
P	3129(1)	602(1)	6022(1)	23(1)
F(1)	4090(2)	583(1)	5283(1)	35(1)
F(2)	3453(2)	1627(1)	6239(1)	31(1)
F(3)	2057(2)	909(1)	5208(1)	30(1)
F(4)	2119(2)	644(1)	6758(1)	36(1)
F(5)	2781(2)	-404(1)	5809(1)	33(1)
F(6)	4176(2)	330(1)	6845(2)	43(1)

Table A2.44. Selected bond lengths [Å] and angles [°] for [Ru(tpm)(bpy)Cl](PF$_6$), **3.10.**

Ru–N(5)	2.022(2)
Ru–N(7)	2.035(2)
Ru–N(8)	2.044(2)
Ru–N(1)	2.063(2)
Ru–N(3)	2.065(2)
Ru–Cl	2.4114(7)
N(5)–Ru–N(7)	90.49(9)
N(5)–Ru–N(8)	92.12(9)
N(7)–Ru–N(8)	79.05(9)
N(5)–Ru–N(1)	85.88(9)
N(7)–Ru–N(1)	99.01(9)
N(8)–Ru–N(1)	177.22(9)
N(5)–Ru–N(3)	87.64(9)
N(7)–Ru–N(3)	176.24(9)
N(8)–Ru–N(3)	97.74(9)
N(1)–Ru–N(3)	84.12(9)
N(5)–Ru–Cl	175.26(6)
N(7)–Ru–Cl	91.49(7)
N(8)–Ru–Cl	92.48(7)
N(1)–Ru–Cl	89.56(7)
N(3)–Ru–Cl	90.62(7)

Symmetry transformations used to generate equivalent atoms:

Table A2.45. Bond lengths [Å] and angles [°] for [Ru(tpm)(bpy)Cl](PF_6), **3.10.**

Ru–N(5)	2.022(2)
Ru–N(7)	2.035(2)
Ru–N(8)	2.044(2)
Ru–N(1)	2.063(2)
Ru–N(3)	2.065(2)
Ru–Cl	2.4114(7)
N(1)–C(3)	1.325(4)
N(1)–N(2)	1.357(3)
N(2)–C(1)	1.354(3)
N(2)–C(10)	1.444(4)
N(3)–C(6)	1.334(4)
N(3)–N(4)	1.357(3)
N(4)–C(4)	1.351(4)
N(4)–C(10)	1.430(4)
N(5)–C(9)	1.326(4)
N(5)–N(6)	1.363(3)
N(6)–C(7)	1.355(4)
N(6)–C(10)	1.449(3)
N(7)–C(11)	1.344(4)
N(7)–C(15)	1.359(4)
N(8)–C(20)	1.334(4)
N(8)–C(16)	1.356(4)
C(1)–C(2)	1.355(4)
C(1)–H(1)	0.9500
C(2)–C(3)	1.395(4)
C(2)–H(2)	0.9500
C(3)–H(3)	0.9500
C(4)–C(5)	1.355(4)
C(4)–H(4)	0.9500
C(5)–C(6)	1.393(4)
C(5)–H(5)	0.9500
C(6)–H(6)	0.9500
C(7)–C(8)	1.362(4)
C(7)–H(7)	0.9500
C(8)–C(9)	1.392(4)
C(8)–H(8)	0.9500
C(9)–H(9)	0.9500
C(10)–H(10)	1.0000
C(11)–C(12)	1.367(4)
C(11)–H(11)	0.9500
C(12)–C(13)	1.375(4)
C(12)–H(12)	0.9500
C(13)–C(14)	1.373(5)
C(13)–H(13)	0.9500
C(14)–C(15)	1.384(4)
C(14)–H(14)	0.9500
C(15)–C(16)	1.463(4)
C(16)–C(17)	1.387(4)
C(17)–C(18)	1.379(5)
C(17)–H(17)	0.9500
C(18)–C(19)	1.375(4)
C(18)–H(18)	0.9500
C(19)–C(20)	1.383(4)
C(19)–H(19)	0.9500
C(20)–H(20)	0.9500
P–F(1)	1.576(2)
P–F(6)	1.582(2)
P–F(5)	1.583(2)

P-F(3)	1.6040(19)
P-F(2)	1.607(2)
P-F(4)	1.612(2)
N(5)-Ru-N(7)	90.49(9)
N(5)-Ru-N(8)	92.12(9)
N(7)-Ru-N(8)	79.05(9)
N(5)-Ru-N(1)	85.88(9)
N(7)-Ru-N(1)	99.01(9)
N(8)-Ru-N(1)	177.22(9)
N(5)-Ru-N(3)	87.64(9)
N(7)-Ru-N(3)	176.24(9)
N(8)-Ru-N(3)	97.74(9)
N(1)-Ru-N(3)	84.12(9)
N(5)-Ru-Cl	175.26(6)
N(7)-Ru-Cl	91.49(7)
N(8)-Ru-Cl	92.48(7)
N(1)-Ru-Cl	89.56(7)
N(3)-Ru-Cl	90.62(7)
C(3)-N(1)-N(2)	104.9(2)
C(3)-N(1)-Ru	137.8(2)
N(2)-N(1)-Ru	117.17(17)
C(1)-N(2)-N(1)	111.5(2)
C(1)-N(2)-C(10)	128.9(2)
N(1)-N(2)-C(10)	119.3(2)
C(6)-N(3)-N(4)	105.0(2)
C(6)-N(3)-Ru	137.6(2)
N(4)-N(3)-Ru	117.32(17)
C(4)-N(4)-N(3)	111.2(2)
C(4)-N(4)-C(10)	129.2(2)
N(3)-N(4)-C(10)	119.2(2)
C(9)-N(5)-N(6)	104.8(2)
C(9)-N(5)-Ru	136.8(2)
N(6)-N(5)-Ru	118.35(17)
C(7)-N(6)-N(5)	111.5(2)
C(7)-N(6)-C(10)	129.6(2)
N(5)-N(6)-C(10)	118.6(2)
C(11)-N(7)-C(15)	117.5(2)
C(11)-N(7)-Ru	126.35(19)
C(15)-N(7)-Ru	116.16(19)
C(20)-N(8)-C(16)	119.0(2)
C(20)-N(8)-Ru	125.6(2)
C(16)-N(8)-Ru	115.42(19)
N(2)-C(1)-C(2)	106.7(3)
N(2)-C(1)-H(1)	126.6
C(2)-C(1)-H(1)	126.6
C(1)-C(2)-C(3)	105.8(3)
C(1)-C(2)-H(2)	127.1
C(3)-C(2)-H(2)	127.1
N(1)-C(3)-C(2)	111.0(3)
N(1)-C(3)-H(3)	124.5
C(2)-C(3)-H(3)	124.5
N(4)-C(4)-C(5)	107.2(3)
N(4)-C(4)-H(4)	126.4
C(5)-C(4)-H(4)	126.4
C(4)-C(5)-C(6)	105.8(3)
C(4)-C(5)-H(5)	127.1
C(6)-C(5)-H(5)	127.1
N(3)-C(6)-C(5)	110.7(3)
N(3)-C(6)-H(6)	124.7
C(5)-C(6)-H(6)	124.7

N(6)-C(7)-C(8)	106.5(3)
N(6)-C(7)-H(7)	126.8
C(8)-C(7)-H(7)	126.8
C(7)-C(8)-C(9)	106.0(3)
C(7)-C(8)-H(8)	127.0
C(9)-C(8)-H(8)	127.0
N(5)-C(9)-C(8)	111.1(3)
N(5)-C(9)-H(9)	124.4
C(8)-C(9)-H(9)	124.4
N(4)-C(10)-N(2)	110.0(2)
N(4)-C(10)-N(6)	111.1(2)
N(2)-C(10)-N(6)	110.0(2)
N(4)-C(10)-H(10)	108.6
N(2)-C(10)-H(10)	108.6
N(6)-C(10)-H(10)	108.6
N(7)-C(11)-C(12)	123.3(3)
N(7)-C(11)-H(11)	118.3
C(12)-C(11)-H(11)	118.3
C(11)-C(12)-C(13)	119.0(3)
C(11)-C(12)-H(12)	120.5
C(13)-C(12)-H(12)	120.5
C(14)-C(13)-C(12)	118.9(3)
C(14)-C(13)-H(13)	120.5
C(12)-C(13)-H(13)	120.5
C(13)-C(14)-C(15)	119.7(3)
C(13)-C(14)-H(14)	120.1
C(15)-C(14)-H(14)	120.1
N(7)-C(15)-C(14)	121.5(3)
N(7)-C(15)-C(16)	114.2(2)
C(14)-C(15)-C(16)	124.3(3)
N(8)-C(16)-C(17)	121.2(3)
N(8)-C(16)-C(15)	115.2(2)
C(17)-C(16)-C(15)	123.7(3)
C(18)-C(17)-C(16)	119.3(3)
C(18)-C(17)-H(17)	120.4
C(16)-C(17)-H(17)	120.4
C(19)-C(18)-C(17)	119.3(3)
C(19)-C(18)-H(18)	120.3
C(17)-C(18)-H(18)	120.3
C(18)-C(19)-C(20)	118.9(3)
C(18)-C(19)-H(19)	120.5
C(20)-C(19)-H(19)	120.5
N(8)-C(20)-C(19)	122.3(3)
N(8)-C(20)-H(20)	118.9
C(19)-C(20)-H(20)	118.9
F(1)-P-F(6)	91.34(12)
F(1)-P-F(5)	90.82(11)
F(6)-P-F(5)	91.45(11)
F(1)-P-F(3)	89.77(11)
F(6)-P-F(3)	177.97(12)
F(5)-P-F(3)	90.23(11)
F(1)-P-F(2)	90.01(11)
F(6)-P-F(2)	89.28(11)
F(5)-P-F(2)	178.87(12)
F(3)-P-F(2)	89.02(11)
F(1)-P-F(4)	178.17(12)
F(6)-P-F(4)	90.27(12)
F(5)-P-F(4)	90.00(11)
F(3)-P-F(4)	88.60(10)
F(2)-P-F(4)	89.14(11)

Symmetry transformations used to generate equivalent atoms:

Table A2.46 Anisotropic displacement parameters (A^2 x 10^3) for [Ru(tpm)(bpy)Cl](PF_6), **3.10.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Ru	13(1)	14(1)	10(1)	1(1)	1(1)	1(1)
N(1)	16(1)	16(1)	13(1)	1(1)	3(1)	1(1)
N(2)	16(1)	19(1)	13(1)	0(1)	1(1)	3(1)
N(3)	15(1)	19(1)	12(1)	0(1)	0(1)	3(1)
N(4)	19(1)	14(1)	16(1)	0(1)	0(1)	3(1)
N(5)	13(1)	18(1)	14(1)	1(1)	-1(1)	2(1)
N(6)	18(1)	18(1)	12(1)	-1(1)	1(1)	4(1)
N(7)	16(1)	17(1)	12(1)	1(1)	3(1)	2(1)
N(8)	15(1)	21(1)	11(1)	0(1)	2(1)	1(1)
Cl	13(1)	14(1)	10(1)	0(1)	3(1)	1(1)
C(1)	15(1)	28(2)	14(1)	2(1)	-1(1)	1(1)
C(2)	20(1)	27(2)	15(1)	-1(1)	0(1)	-1(1)
C(3)	21(1)	21(2)	15(1)	-2(1)	3(1)	-3(1)
C(4)	32(2)	14(1)	16(1)	-1(1)	-3(1)	1(1)
C(5)	32(2)	17(2)	17(1)	1(1)	0(1)	-6(1)
C(6)	21(1)	22(2)	12(1)	-1(1)	0(1)	-5(1)
C(7)	18(1)	25(2)	16(1)	-5(1)	4(1)	3(1)
C(8)	22(2)	29(2)	13(1)	-1(1)	1(1)	2(1)
C(9)	19(1)	24(2)	14(1)	1(1)	0(1)	1(1)
C(10)	19(1)	18(1)	13(1)	1(1)	-1(1)	2(1)
C(11)	18(1)	22(2)	22(2)	2(1)	5(1)	0(1)
C(12)	26(2)	18(2)	31(2)	3(1)	10(1)	-2(1)
C(13)	30(2)	21(2)	28(2)	8(1)	13(1)	5(1)
C(14)	23(2)	24(2)	20(2)	6(1)	7(1)	8(1)
C(15)	18(1)	21(2)	14(1)	1(1)	7(1)	4(1)
C(16)	20(1)	22(2)	12(1)	0(1)	5(1)	5(1)
C(17)	22(2)	32(2)	13(1)	4(1)	1(1)	8(1)
C(18)	19(1)	36(2)	14(1)	-1(1)	-1(1)	4(1)
C(19)	20(1)	32(2)	16(1)	-4(1)	1(1)	-3(1)
C(20)	19(1)	24(2)	14(1)	-1(1)	2(1)	0(1)
P	25(1)	22(1)	22(1)	-1(1)	-3(1)	4(1)
F(1)	28(1)	36(1)	42(1)	-10(1)	8(1)	3(1)
F(2)	38(1)	23(1)	32(1)	-3(1)	5(1)	2(1)
F(3)	28(1)	37(1)	24(1)	2(1)	-1(1)	13(1)
F(4)	45(1)	36(1)	27(1)	1(1)	10(1)	-4(1)
F(5)	43(1)	23(1)	29(1)	-2(1)	-6(1)	1(1)
F(6)	47(1)	33(1)	41(1)	1(1)	-20(1)	3(1)

Table A2.47. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for [Ru(tpm)(bpy)Cl](PF_6), **3.10.**

	x	y	z	U(eq)
H(1)	11836	2234	7023	23
H(2)	11596	3619	7936	25
H(3)	9496	4240	7337	22
H(4)	9211	108	6372	25
H(5)	7070	-55	6814	27
H(6)	6004	1407	6477	23
H(7)	10729	1605	3947	23
H(8)	9717	2578	2655	26
H(9)	8059	3434	3327	23
H(10)	10426	1390	5717	21
H(11)	9094	4905	5723	24
H(12)	8874	6323	5135	29
H(13)	7066	6707	4143	31
H(14)	5562	5621	3725	26
H(17)	4266	4523	3377	27
H(18)	2982	3279	3093	28
H(19)	3568	1946	3855	27
H(20)	5423	1879	4868	23

Table A2.48. Hydrogen bonds for [Ru(tpm)(bpy)Cl](PF_6), **3.10** [Å] and angles [°].

D–H...A	d(D–H)	d(H...A)	d(D...A)	<(DHA)

Table A2.49. Crystal data and structure refinement for $[Ru(tpm)(bpy)(H_2O)](ClO_4)_2.MeOH$, **3.11.**

Identification code	**3.11**
Empirical formula	C21 H24 Cl2 N8 O10 Ru
Formula weight	720.45
Temperature	113(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, *p* 21/*c*
Unit cell dimensions	a = 14.9764(17) Å alpha = 90°. b = 14.4923(18) Å beta = 116.902(2)°. c = 14.3878(16) Å gamma = 90°.
Volume	2784.8(6) $Å^3$
Z, Calculated density	4, 1.718 Mg/m^3
Absorption coefficient	0.824 mm^{-1}
F(000)	1456
Crystal size	0.49 x 0.18 x 0.15 mm
Theta range for data collection	2.07 to 26.38°.
Limiting indices	-17<=h<=18, -18<=k<=14, -17<=l<=17
Reflections collected / unique	16052 / 5653 [R(int) = 0.0444]
Completeness to theta = 26.38	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.8864 and 0.6884
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5653 / 0 / 382
Goodness-of-fit on F^2	1.037
Final R indices [I>2sigma(I)]	R1 = 0.0445, wR2 = 0.1078
R indices (all data)	R1 = 0.0720, wR2 = 0.1183
Largest diff. peak and hole	1.322 and -1.055 eA^{-3}

Table A2.50. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for [Ru(tpm)(bpy)(H_2O)](ClO_4)$_2$.MeOH, **3.11.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ru	2752(1)	6378(1)	551(1)	22(1)
N(1)	2022(3)	6666(2)	1449(3)	27(1)
N(2)	1366(3)	7386(2)	1142(3)	24(1)
N(3)	1302(3)	6421(2)	-651(3)	24(1)
N(4)	717(2)	7156(2)	-680(3)	23(1)
N(5)	2819(2)	7747(2)	365(3)	23(1)
N(6)	1996(3)	8278(2)	169(3)	22(1)
N(7)	4203(3)	6349(2)	1699(3)	28(1)
N(8)	3511(3)	6187(2)	-317(3)	26(1)
C(1)	852(3)	7428(3)	1710(3)	31(1)
C(2)	1193(4)	6736(3)	2417(4)	35(1)
C(3)	1914(4)	6275(3)	2231(3)	33(1)
C(4)	-250(3)	7028(3)	-1410(3)	31(1)
C(5)	-287(4)	6212(3)	-1893(4)	35(1)
C(6)	686(3)	5856(3)	-1401(4)	31(1)
C(7)	2166(3)	9183(3)	31(3)	24(1)
C(8)	3117(3)	9230(3)	146(3)	28(1)
C(9)	3503(3)	8333(3)	355(3)	27(1)
C(10)	1124(3)	7838(3)	154(3)	22(1)
C(11)	4502(4)	6434(3)	2729(4)	35(1)
C(12)	5493(4)	6394(3)	3460(4)	41(1)
C(13)	6216(4)	6267(3)	3117(4)	43(1)
C(14)	5921(4)	6189(3)	2071(4)	35(1)
C(15)	4911(3)	6233(3)	1368(4)	30(1)
C(16)	4528(3)	6194(3)	227(4)	27(1)
C(17)	5130(4)	6179(3)	-274(4)	34(1)
C(18)	4687(4)	6163(3)	-1356(4)	38(1)
C(19)	3658(4)	6151(3)	-1902(4)	35(1)
C(20)	3097(4)	6175(3)	-1367(3)	29(1)
O(1)	2644(3)	4920(2)	762(2)	41(1)
Cl(1)	3339(1)	3624(1)	3176(1)	39(1)
O(11)	4088(3)	3152(3)	4038(3)	49(1)
O(12)	2642(3)	2986(3)	2453(4)	85(2)
O(13)	3803(4)	4117(5)	2668(4)	111(2)
O(14)	2800(3)	4220(3)	3507(4)	78(2)
Cl(2)	1007(1)	4539(1)	6525(1)	25(1)
O(21)	1800(3)	4208(3)	7481(3)	52(1)
O(22)	75(2)	4379(2)	6542(3)	42(1)
O(23)	1016(3)	4050(2)	5656(3)	43(1)
O(24)	1155(3)	5505(2)	6446(3)	41(1)
C(99)	2003(6)	2251(5)	4681(6)	90(2)
O(99)	2249(7)	1376(4)	4413(4)	130(3)

Table A2.51. Selected bond lengths [Å] and angles [°] for $[Ru(tpm)(bpy)(H_2O)](ClO_4)_2.MeOH$, **3.11.**

Ru–N(5)	2.011(3)
Ru–N(7)	2.051(4)
Ru–N(8)	2.053(3)
Ru–N(3)	2.076(4)
Ru–N(1)	2.077(4)
Ru–O(1)	2.151(3)
N(5)–Ru–N(7)	91.63(14)
N(5)–Ru–N(8)	88.69(13)
N(7)–Ru–N(8)	79.06(14)
N(5)–Ru–N(3)	87.49(13)
N(7)–Ru–N(3)	177.87(14)
N(8)–Ru–N(3)	98.97(14)
N(5)–Ru–N(1)	87.51(14)
N(7)–Ru–N(1)	99.63(14)
N(8)–Ru–N(1)	175.94(14)
N(3)–Ru–N(1)	82.28(13)
N(5)–Ru–O(1)	178.29(13)
N(7)–Ru–O(1)	89.22(13)
N(8)–Ru–O(1)	92.93(13)
N(3)–Ru–O(1)	91.70(13)
N(1)–Ru–O(1)	90.89(13)

Symmetry transformations used to generate equivalent atoms:

Table A2.52. Bond lengths [Å] and angles [°] for $[Ru(tpm)(bpy)(H_2O)](ClO_4)_2.MeOH$, **3.11.**

Ru–N(5)	2.011(3)
Ru–N(7)	2.051(4)
Ru–N(8)	2.053(3)
Ru–N(3)	2.076(4)
Ru–N(1)	2.077(4)
Ru–O(1)	2.151(3)
N(1)–C(3)	1.334(5)
N(1)–N(2)	1.362(5)
N(2)–C(1)	1.354(5)
N(2)–C(10)	1.454(5)
N(3)–C(6)	1.336(6)
N(3)–N(4)	1.369(5)
N(4)–C(4)	1.364(5)
N(4)–C(10)	1.459(5)
N(5)–C(9)	1.335(5)
N(5)–N(6)	1.369(5)
N(6)–C(7)	1.368(5)
N(6)–C(10)	1.444(5)
N(7)–C(11)	1.345(6)
N(7)–C(15)	1.356(6)
N(8)–C(20)	1.349(5)
N(8)–C(16)	1.361(6)
C(1)–C(2)	1.353(6)
C(1)–H(1)	0.9300
C(2)–C(3)	1.394(7)
C(2)–H(2)	0.9300
C(3)–H(3)	0.9300
C(4)–C(5)	1.360(6)
C(4)–H(4)	0.9300
C(5)–C(6)	1.398(6)
C(5)–H(5)	0.9300
C(6)–H(6)	0.9300
C(7)–C(8)	1.360(6)
C(7)–H(7)	0.9300
C(8)–C(9)	1.398(6)
C(8)–H(8)	0.9300
C(9)–H(9)	0.9300
C(10)–H(10)	0.9800
C(11)–C(12)	1.378(7)
C(11)–H(11)	0.9300
C(12)–C(13)	1.391(7)
C(12)–H(12)	0.9300
C(13)–C(14)	1.367(7)
C(13)–H(13)	0.9300
C(14)–C(15)	1.390(6)
C(14)–H(14)	0.9300
C(15)–C(16)	1.475(6)
C(16)–C(17)	1.386(6)
C(17)–C(18)	1.388(7)
C(17)–H(17)	0.9300
C(18)–C(19)	1.377(7)
C(18)–H(18)	0.9300
C(19)–C(20)	1.373(6)
C(19)–H(19)	0.9300
C(20)–H(20)	0.9300
O(1)–H(1A)	0.8200
O(1)–H(1B)	0.5982

Cl(1)-O(14)	1.406(4)
Cl(1)-O(11)	1.416(4)
Cl(1)-O(13)	1.410(5)
Cl(1)-O(12)	1.430(4)
Cl(2)-O(22)	1.425(3)
Cl(2)-O(24)	1.430(3)
Cl(2)-O(21)	1.434(4)
Cl(2)-O(23)	1.442(3)
C(99)-O(99)	1.422(9)
C(99)-H(99A)	0.9600
C(99)-H(99B)	0.9600
C(99)-H(99C)	0.9600
O(99)-H(99)	0.8200

N(5)-Ru-N(7)	91.63(14)
N(5)-Ru-N(8)	88.69(13)
N(7)-Ru-N(8)	79.06(14)
N(5)-Ru-N(3)	87.49(13)
N(7)-Ru-N(3)	177.87(14)
N(8)-Ru-N(3)	98.97(14)
N(5)-Ru-N(1)	87.51(14)
N(7)-Ru-N(1)	99.63(14)
N(8)-Ru-N(1)	175.94(14)
N(3)-Ru-N(1)	82.28(13)
N(5)-Ru-O(1)	178.29(13)
N(7)-Ru-O(1)	89.22(13)
N(8)-Ru-O(1)	92.93(13)
N(3)-Ru-O(1)	91.70(13)
N(1)-Ru-O(1)	90.89(13)
C(3)-N(1)-N(2)	104.7(4)
C(3)-N(1)-Ru	137.6(3)
N(2)-N(1)-Ru	117.2(2)
C(1)-N(2)-N(1)	111.2(3)
C(1)-N(2)-C(10)	127.7(4)
N(1)-N(2)-C(10)	119.1(3)
C(6)-N(3)-N(4)	104.7(3)
C(6)-N(3)-Ru	137.3(3)
N(4)-N(3)-Ru	117.6(3)
C(4)-N(4)-N(3)	111.1(3)
C(4)-N(4)-C(10)	129.0(3)
N(3)-N(4)-C(10)	118.6(3)
C(9)-N(5)-N(6)	105.3(3)
C(9)-N(5)-Ru	135.5(3)
N(6)-N(5)-Ru	119.1(3)
C(7)-N(6)-N(5)	111.1(3)
C(7)-N(6)-C(10)	130.3(3)
N(5)-N(6)-C(10)	118.6(3)
C(11)-N(7)-C(15)	118.4(4)
C(11)-N(7)-Ru	126.1(3)
C(15)-N(7)-Ru	115.6(3)
C(20)-N(8)-C(16)	118.2(4)
C(20)-N(8)-Ru	125.6(3)
C(16)-N(8)-Ru	115.6(3)
N(2)-C(1)-C(2)	107.2(4)
N(2)-C(1)-H(1)	126.4
C(2)-C(1)-H(1)	126.4
C(1)-C(2)-C(3)	105.8(4)
C(1)-C(2)-H(2)	127.1
C(3)-C(2)-H(2)	127.1
N(1)-C(3)-C(2)	111.0(4)
N(1)-C(3)-H(3)	124.5

C(2)-C(3)-H(3)	124.5
C(5)-C(4)-N(4)	107.2(4)
C(5)-C(4)-H(4)	126.4
N(4)-C(4)-H(4)	126.4
C(4)-C(5)-C(6)	105.7(4)
C(4)-C(5)-H(5)	127.2
C(6)-C(5)-H(5)	127.2
N(3)-C(6)-C(5)	111.4(4)
N(3)-C(6)-H(6)	124.3
C(5)-C(6)-H(6)	124.3
N(6)-C(7)-C(8)	106.4(4)
N(6)-C(7)-H(7)	126.8
C(8)-C(7)-H(7)	126.8
C(7)-C(8)-C(9)	106.8(4)
C(7)-C(8)-H(8)	126.6
C(9)-C(8)-H(8)	126.6
N(5)-C(9)-C(8)	110.4(4)
N(5)-C(9)-H(9)	124.8
C(8)-C(9)-H(9)	124.8
N(6)-C(10)-N(2)	111.1(3)
N(6)-C(10)-N(4)	110.5(3)
N(2)-C(10)-N(4)	109.0(3)
N(6)-C(10)-H(10)	108.7
N(2)-C(10)-H(10)	108.7
N(4)-C(10)-H(10)	108.7
N(7)-C(11)-C(12)	122.9(5)
N(7)-C(11)-H(11)	118.5
C(12)-C(11)-H(11)	118.5
C(11)-C(12)-C(13)	118.5(5)
C(11)-C(12)-H(12)	120.8
C(13)-C(12)-H(12)	120.8
C(14)-C(13)-C(12)	119.1(5)
C(14)-C(13)-H(13)	120.5
C(12)-C(13)-H(13)	120.5
C(13)-C(14)-C(15)	120.0(5)
C(13)-C(14)-H(14)	120.0
C(15)-C(14)-H(14)	120.0
N(7)-C(15)-C(14)	121.1(4)
N(7)-C(15)-C(16)	115.1(4)
C(14)-C(15)-C(16)	123.8(4)
N(8)-C(16)-C(17)	121.5(4)
N(8)-C(16)-C(15)	114.3(4)
C(17)-C(16)-C(15)	124.2(4)
C(16)-C(17)-C(18)	119.3(5)
C(16)-C(17)-H(17)	120.4
C(18)-C(17)-H(17)	120.4
C(19)-C(18)-C(17)	119.0(4)
C(19)-C(18)-H(18)	120.5
C(17)-C(18)-H(18)	120.5
C(18)-C(19)-C(20)	119.4(5)
C(18)-C(19)-H(19)	120.3
C(20)-C(19)-H(19)	120.3
N(8)-C(20)-C(19)	122.7(4)
N(8)-C(20)-H(20)	118.7
C(19)-C(20)-H(20)	118.7
Ru-O(1)-H(1A)	109.5
Ru-O(1)-H(1B)	123.0
H(1A)-O(1)-H(1B)	127.0
O(14)-Cl(1)-O(11)	110.4(3)
O(14)-Cl(1)-O(13)	111.0(4)
O(11)-Cl(1)-O(13)	108.6(3)

O(14)-Cl(1)-O(12)	107.6(3)
O(11)-Cl(1)-O(12)	110.7(3)
O(13)-Cl(1)-O(12)	108.6(4)
O(22)-Cl(2)-O(24)	110.4(2)
O(22)-Cl(2)-O(21)	108.7(2)
O(24)-Cl(2)-O(21)	108.2(2)
O(22)-Cl(2)-O(23)	109.2(2)
O(24)-Cl(2)-O(23)	110.5(2)
O(21)-Cl(2)-O(23)	109.8(2)
O(99)-C(99)-H(99A)	109.5
O(99)-C(99)-H(99B)	109.5
H(99A)-C(99)-H(99B)	109.5
O(99)-C(99)-H(99C)	109.5
H(99A)-C(99)-H(99C)	109.5
H(99B)-C(99)-H(99C)	109.5
C(99)-O(99)-H(99)	109.5

Symmetry transformations used to generate equivalent atoms:

Table A2.53. Anisotropic displacement parameters (A^2 x 10^3) for [Ru(tpm)(bpy)(H_2O)](ClO_4)$_2$.MeOH, **3.11.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2$ [$h^2\ a^{*2}$ U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Ru	24(1)	20(1)	21(1)	2(1)	11(1)	4(1)
N(1)	31(2)	27(2)	22(2)	2(2)	11(2)	1(2)
N(2)	27(2)	23(2)	24(2)	0(2)	14(2)	3(2)
N(3)	25(2)	23(2)	26(2)	-1(2)	15(2)	2(2)
N(4)	22(2)	23(2)	23(2)	0(1)	11(2)	5(2)
N(5)	24(2)	24(2)	23(2)	2(1)	12(2)	4(2)
N(6)	23(2)	23(2)	20(2)	1(1)	10(2)	4(2)
N(7)	31(2)	23(2)	28(2)	3(2)	12(2)	6(2)
N(8)	25(2)	22(2)	30(2)	-2(2)	13(2)	4(2)
C(1)	37(3)	31(3)	34(2)	-7(2)	24(2)	2(2)
C(2)	48(3)	39(3)	31(2)	2(2)	28(2)	0(2)
C(3)	49(3)	29(3)	25(2)	6(2)	20(2)	0(2)
C(4)	25(2)	32(3)	32(2)	1(2)	11(2)	3(2)
C(5)	26(2)	42(3)	32(2)	-4(2)	9(2)	-2(2)
C(6)	33(3)	28(2)	32(2)	-7(2)	15(2)	-6(2)
C(7)	32(2)	18(2)	21(2)	1(2)	11(2)	2(2)
C(8)	34(3)	21(2)	30(2)	-4(2)	14(2)	-7(2)
C(9)	27(2)	32(2)	25(2)	-3(2)	13(2)	-2(2)
C(10)	23(2)	20(2)	23(2)	0(2)	11(2)	5(2)
C(11)	37(3)	35(3)	28(2)	4(2)	10(2)	9(2)
C(12)	45(3)	35(3)	28(2)	4(2)	2(2)	7(2)
C(13)	31(3)	34(3)	48(3)	4(2)	3(2)	4(2)
C(14)	27(3)	30(3)	42(3)	3(2)	11(2)	6(2)
C(15)	29(2)	19(2)	38(3)	2(2)	13(2)	6(2)
C(16)	31(2)	15(2)	37(3)	1(2)	18(2)	6(2)
C(17)	31(3)	26(3)	49(3)	-5(2)	22(2)	-2(2)
C(18)	43(3)	33(3)	52(3)	-11(2)	34(3)	-8(2)
C(19)	44(3)	33(3)	37(3)	-5(2)	27(2)	-2(2)
C(20)	32(3)	28(2)	31(2)	-3(2)	17(2)	3(2)
O(1)	56(3)	27(2)	31(2)	3(1)	13(2)	-2(2)
Cl(1)	42(1)	40(1)	33(1)	6(1)	15(1)	8(1)
O(11)	40(2)	57(2)	40(2)	13(2)	12(2)	9(2)
O(12)	44(3)	80(3)	94(4)	-51(3)	-1(3)	3(2)
O(13)	53(3)	182(6)	88(4)	77(4)	23(3)	-10(3)
O(14)	64(3)	66(3)	83(3)	-22(2)	15(3)	25(2)
Cl(2)	26(1)	24(1)	26(1)	0(1)	13(1)	-5(1)
O(21)	38(2)	59(3)	45(2)	14(2)	6(2)	10(2)
O(22)	34(2)	49(2)	48(2)	-13(2)	23(2)	-17(2)
O(23)	58(2)	40(2)	47(2)	-17(2)	38(2)	-18(2)
O(24)	58(2)	26(2)	44(2)	0(2)	28(2)	-10(2)
C(99)	113(7)	71(5)	80(5)	-6(4)	37(5)	10(5)
O(99)	233(9)	73(4)	66(4)	1(3)	53(5)	12(4)

Table A2.54. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for [Ru(tpm)(bpy)(H_2O)](ClO_4)$_2$.MeOH, **3.11.**

	x	y	z	U(eq)
H(1)	356	7852	1629	37
H(2)	989	6596	2922	42
H(3)	2273	5763	2604	40
H(4)	-785	7425	-1552	37
H(5)	-843	5945	-2436	42
H(6)	879	5298	-1573	37
H(7)	1719	9671	-113	29
H(8)	3448	9756	96	34
H(9)	4145	8168	471	33
H(10)	613	8310	30	27
H(11)	4018	6524	2957	42
H(12)	5674	6450	4167	49
H(13)	6890	6236	3592	52
H(14)	6397	6106	1831	42
H(17)	5824	6179	110	41
H(18)	5078	6161	-1706	45
H(19)	3346	6128	-2626	42
H(20)	2403	6182	-1744	35
H(1A)	2256	4687	205	61
H(1B)	2906	4723	1158	61
H(99A)	1967	2204	5329	136
H(99B)	1367	2450	4144	136
H(99C)	2509	2692	4752	136
H(99)	2097	1367	3790	194

Table A2.55. Hydrogen bonds for [Ru(tpm)(bpy)(H_2O)](ClO_4)$_2$.MeOH, **3.11** [Å and °].

D–H...A	d(D–H)	d(H...A)	d(D...A)	<(DHA)
O(99)–H(99)...O(21)#1	0.82	1.92	2.684(6)	155.2
O(1)–H(1B)...O(13)	0.60	2.16	2.753(6)	174.1
O(1)–H(1A)...O(99)#1	0.82	1.91	2.570(6)	136.4

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z-1/2

Table A2.56. Crystal data and structure refinement for [Ru(ttp)(bpy)Cl](PF$_6$), **3.13.**

Identification code	**3.13**
Empirical formula	C32 H25 Cl F6 N5 P Ru
Formula weight	761.06
Temperature	93(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, *p* 21/*c*
Unit cell dimensions	a = 13.0766(15) Å alpha = 90°. b = 19.140(3) Å beta = 100.049(2)°. c = 12.2565(16) Å gamma = 90°.
Volume	3020.6(7) Å^3
Z, Calculated density	4, 1.674 Mg/m^3
Absorption coefficient	0.731 mm^{-1}
F(000)	1528
Crystal size	0.46 x 0.10 x 0.06 mm
Theta range for data collection	1.99 to 25.05 deg.
Limiting indices	-15<=h<=15, -22<=k<=22, -13<=l<=14
Reflections collected / unique	21587 / 5207 [R(int) = 0.0692]
Completeness to theta = 25.05	97.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9575 and 0.7298
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5207 / 39 / 481
Goodness-of-fit on F^2	1.010
Final R indices [I>2sigma(I)]	R1 = 0.0356, wR2 = 0.0692
R indices (all data)	R1 = 0.0652, wR2 = 0.0769
Largest diff. peak and hole	0.500 and -0.491 eÅ^{-3}

Table A2.57. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for [Ru(ttp)(bpy)Cl](PF_6), **3.13.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ru	7540(1)	10467(1)	6999(1)	13(1)
N(1)	8827(2)	9994(1)	6560(2)	12(1)
N(2)	6920(2)	9620(2)	6291(2)	17(1)
N(3)	6002(2)	10638(2)	7112(2)	22(1)
N(4)	7942(2)	10039(1)	8533(2)	16(1)
N(5)	8131(2)	11329(1)	7933(2)	18(1)
C(1)	9796(2)	10247(2)	6693(3)	16(1)
C(2)	10611(2)	9875(2)	6392(3)	17(1)
C(3)	10417(3)	9218(2)	5927(3)	18(1)
C(4)	9417(3)	8954(2)	5775(3)	17(1)
C(5)	8628(2)	9348(2)	6085(3)	14(1)
C(6)	7531(3)	9124(2)	5933(3)	16(1)
C(7)	7107(3)	8496(2)	5521(3)	22(1)
C(8)	6048(3)	8365(2)	5470(3)	31(1)
C(9)	5438(3)	8891(2)	5830(3)	32(1)
C(10)	5884(3)	9513(2)	6232(3)	23(1)
C(11)	5352(3)	10109(2)	6650(3)	28(1)
C(12)	4292(3)	10159(3)	6589(3)	45(1)
C(13)	3867(3)	10740(3)	6999(4)	60(2)
C(14)	4512(3)	11268(3)	7463(4)	53(2)
C(15)	5568(3)	11203(2)	7495(3)	34(1)
C(16)	5668(8)	7569(5)	5227(10)	18(3)
C(17)	6312(5)	6979(3)	5271(6)	19(2)
C(18)	5887(5)	6326(3)	5048(6)	21(2)
C(19)	4825(5)	6229(4)	4745(6)	19(2)
C(20)	4190(10)	6812(7)	4659(19)	20(4)
C(21)	4601(10)	7477(7)	4886(11)	17(3)
C(22)	4342(6)	5507(4)	4591(7)	34(2)
C(16')	5460(8)	7789(5)	4930(8)	16(2)
C(17')	5842(5)	7398(3)	4127(6)	20(2)
C(18')	5291(5)	6821(3)	3662(6)	25(2)
C(19')	4374(5)	6601(4)	3968(7)	24(2)
C(20')	4017(12)	6989(8)	4789(19)	20(3)
C(21')	4545(9)	7575(7)	5261(11)	14(3)
C(22')	3768(6)	5977(4)	3433(7)	40(2)
C(23)	8401(2)	10473(2)	9359(3)	18(1)
C(24)	8763(3)	10217(2)	10415(3)	26(1)
C(25)	8685(4)	9515(2)	10632(3)	44(1)
C(26)	8251(4)	9079(2)	9791(4)	50(1)
C(27)	7886(3)	9350(2)	8762(3)	30(1)
C(28)	8480(2)	11202(2)	9032(3)	17(1)
C(29)	8835(3)	11738(2)	9765(3)	24(1)
C(30)	8806(3)	12418(2)	9379(3)	35(1)
C(31)	8449(4)	12544(2)	8278(4)	43(1)
C(32)	8120(3)	11994(2)	7577(3)	30(1)
Cl	7304(1)	11077(1)	5253(1)	20(1)
P	8768(1)	7296(1)	7918(1)	27(1)
F(1)	8704(2)	7427(1)	9201(2)	39(1)
F(2)	9269(2)	8051(1)	7861(2)	34(1)
F(3)	7636(2)	7617(2)	7585(2)	55(1)
F(4)	9904(2)	6973(1)	8285(2)	42(1)
F(5)	8296(2)	6526(1)	8007(2)	61(1)
F(6)	8836(2)	7163(1)	6652(2)	41(1)

Table A2.58. Selected bond lengths [Å] and angles [°] for [Ru(ttp)(bpy)Cl](PF_6), **3.13.**

Ru–N(2)	1.949(3)
Ru–N(4)	2.033(3)
Ru–N(1)	2.064(3)
Ru–N(3)	2.065(3)
Ru–N(5)	2.079(3)
Ru–Cl	2.4103(9)
N(2)–Ru–N(4)	95.56(11)
N(2)–Ru–N(1)	79.24(10)
N(4)–Ru–N(1)	88.47(10)
N(2)–Ru–N(3)	80.05(11)
N(4)–Ru–N(3)	95.71(11)
N(1)–Ru–N(3)	159.17(11)
N(2)–Ru–N(5)	172.73(11)
N(4)–Ru–N(5)	78.44(11)
N(1)–Ru–N(5)	104.48(11)
N(3)–Ru–N(5)	96.35(11)
N(2)–Ru–Cl	91.63(8)
N(4)–Ru–Cl	171.24(8)
N(1)–Ru–Cl	87.96(7)
N(3)–Ru–Cl	90.45(8)
N(5)–Ru–Cl	94.73(8)

Symmetry transformations used to generate equivalent atoms:

Table A2.59. Bond lengths [Å] and angles [°] for [Ru(ttp)(bpy)Cl](PF_6), **3.13.**

Ru–N(2)	1.949(3)
Ru–N(4)	2.033(3)
Ru–N(1)	2.064(3)
Ru–N(3)	2.065(3)
Ru–N(5)	2.079(3)
Ru–Cl	2.4103(9)
N(1)–C(1)	1.340(4)
N(1)–C(5)	1.373(4)
N(2)–C(10)	1.359(4)
N(2)–C(6)	1.362(4)
N(3)–C(15)	1.343(5)
N(3)–C(11)	1.378(5)
N(4)–C(27)	1.354(4)
N(4)–C(23)	1.364(4)
N(5)–C(32)	1.344(4)
N(5)–C(28)	1.366(4)
C(1)–C(2)	1.385(5)
C(1)–H(1)	0.9500
C(2)–C(3)	1.385(5)
C(2)–H(2)	0.9500
C(3)–C(4)	1.383(5)
C(3)–H(3)	0.9500
C(4)–C(5)	1.383(4)
C(4)–H(4)	0.9500
C(5)–C(6)	1.478(4)
C(6)–C(7)	1.381(5)
C(7)–C(8)	1.398(5)
C(7)–H(7)	0.9500
C(8)–C(9)	1.402(6)
C(8)–C(16')	1.437(11)
C(8)–C(16)	1.613(12)
C(9)–C(10)	1.379(5)
C(9)–H(9)	0.9500
C(10)–C(11)	1.475(5)
C(11)–C(12)	1.379(5)
C(12)–C(13)	1.376(6)
C(12)–H(12)	0.9500
C(13)–C(14)	1.375(6)
C(13)–H(13)	0.9500
C(14)–C(15)	1.381(5)
C(14)–H(14)	0.9500
C(15)–H(15)	0.9500
C(16)–C(21)	1.397(12)
C(16)–C(17)	1.404(10)
C(17)–C(18)	1.376(8)
C(17)–H(17)	0.9500
C(18)–C(19)	1.386(8)
C(18)–H(18)	0.9500
C(19)–C(20)	1.384(13)
C(19)–C(22)	1.518(9)
C(20)–C(21)	1.391(12)
C(20)–H(20)	0.9500
C(21)–H(21)	0.9500
C(22)–H(22A)	0.9800
C(22)–H(22B)	0.9800
C(22)–H(22C)	0.9800

C(16')-C(21')	1.391(10)
C(16')-C(17')	1.396(10)
C(17')-C(18')	1.386(8)
C(17')-H(17')	0.9500
C(18')-C(19')	1.383(9)
C(18')-H(18')	0.9500
C(19')-C(20')	1.395(13)
C(19')-C(22')	1.516(9)
C(20')-C(21')	1.390(12)
C(20')-H(20')	0.9500
C(21')-H(21')	0.9500
C(22')-H(22D)	0.9800
C(22')-H(22E)	0.9800
C(22')-H(22F)	0.9800
C(23)-C(24)	1.388(5)
C(23)-C(28)	1.462(5)
C(24)-C(25)	1.376(5)
C(24)-H(24)	0.9500
C(25)-C(26)	1.370(6)
C(25)-H(25)	0.9500
C(26)-C(27)	1.370(6)
C(26)-H(26)	0.9500
C(27)-H(27)	0.9500
C(28)-C(29)	1.389(5)
C(29)-C(30)	1.382(5)
C(29)-H(29)	0.9500
C(30)-C(31)	1.371(6)
C(30)-H(30)	0.9500
C(31)-C(32)	1.380(5)
C(31)-H(31)	0.9500
C(32)-H(32)	0.9500
P-F(3)	1.589(3)
P-F(6)	1.590(2)
P-F(2)	1.593(2)
P-F(4)	1.599(2)
P-F(5)	1.608(2)
P-F(1)	1.610(2)

N(2)-Ru-N(4)	95.56(11)
N(2)-Ru-N(1)	79.24(10)
N(4)-Ru-N(1)	88.47(10)
N(2)-Ru-N(3)	80.05(11)
N(4)-Ru-N(3)	95.71(11)
N(1)-Ru-N(3)	159.17(11)
N(2)-Ru-N(5)	172.73(11)
N(4)-Ru-N(5)	78.44(11)
N(1)-Ru-N(5)	104.48(11)
N(3)-Ru-N(5)	96.35(11)
N(2)-Ru-Cl	91.63(8)
N(4)-Ru-Cl	171.24(8)
N(1)-Ru-Cl	87.96(7)
N(3)-Ru-Cl	90.45(8)
N(5)-Ru-Cl	94.73(8)
C(1)-N(1)-C(5)	118.8(3)
C(1)-N(1)-Ru	127.4(2)
C(5)-N(1)-Ru	113.8(2)
C(10)-N(2)-C(6)	120.9(3)
C(10)-N(2)-Ru	118.8(2)
C(6)-N(2)-Ru	120.1(2)
C(15)-N(3)-C(11)	117.8(3)
C(15)-N(3)-Ru	128.8(3)

C(11)-N(3)-Ru	113.2(2)
C(27)-N(4)-C(23)	118.1(3)
C(27)-N(4)-Ru	124.7(2)
C(23)-N(4)-Ru	116.8(2)
C(32)-N(5)-C(28)	118.1(3)
C(32)-N(5)-Ru	126.2(3)
C(28)-N(5)-Ru	115.4(2)
N(1)-C(1)-C(2)	122.6(3)
N(1)-C(1)-H(1)	118.7
C(2)-C(1)-H(1)	118.7
C(3)-C(2)-C(1)	118.8(3)
C(3)-C(2)-H(2)	120.6
C(1)-C(2)-H(2)	120.6
C(4)-C(3)-C(2)	119.2(3)
C(4)-C(3)-H(3)	120.4
C(2)-C(3)-H(3)	120.4
C(5)-C(4)-C(3)	119.8(3)
C(5)-C(4)-H(4)	120.1
C(3)-C(4)-H(4)	120.1
N(1)-C(5)-C(4)	120.8(3)
N(1)-C(5)-C(6)	115.0(3)
C(4)-C(5)-C(6)	124.2(3)
N(2)-C(6)-C(7)	120.2(3)
N(2)-C(6)-C(5)	111.6(3)
C(7)-C(6)-C(5)	128.1(3)
C(6)-C(7)-C(8)	120.3(3)
C(6)-C(7)-H(7)	119.9
C(8)-C(7)-H(7)	119.9
C(7)-C(8)-C(9)	118.1(3)
C(7)-C(8)-C(16')	127.2(5)
C(9)-C(8)-C(16')	114.0(5)
C(7)-C(8)-C(16)	116.6(5)
C(9)-C(8)-C(16)	124.2(5)
C(16')-C(8)-C(16)	21.4(4)
C(10)-C(9)-C(8)	120.2(3)
C(10)-C(9)-H(9)	119.9
C(8)-C(9)-H(9)	119.9
N(2)-C(10)-C(9)	120.3(3)
N(2)-C(10)-C(11)	113.0(3)
C(9)-C(10)-C(11)	126.7(3)
N(3)-C(11)-C(12)	120.9(4)
N(3)-C(11)-C(10)	114.6(3)
C(12)-C(11)-C(10)	124.4(4)
C(13)-C(12)-C(11)	120.1(4)
C(13)-C(12)-H(12)	119.9
C(11)-C(12)-H(12)	119.9
C(12)-C(13)-C(14)	119.2(4)
C(12)-C(13)-H(13)	120.4
C(14)-C(13)-H(13)	120.4
C(13)-C(14)-C(15)	118.9(4)
C(13)-C(14)-H(14)	120.6
C(15)-C(14)-H(14)	120.6
N(3)-C(15)-C(14)	123.0(4)
N(3)-C(15)-H(15)	118.5
C(14)-C(15)-H(15)	118.5
C(21)-C(16)-C(17)	118.1(10)
C(21)-C(16)-C(8)	115.7(8)
C(17)-C(16)-C(8)	126.1(8)
C(18)-C(17)-C(16)	120.3(7)
C(18)-C(17)-H(17)	119.8
C(16)-C(17)-H(17)	119.8

C(17)-C(18)-C(19)	121.7(6)
C(17)-C(18)-H(18)	119.2
C(19)-C(18)-H(18)	119.2
C(20)-C(19)-C(18)	118.3(8)
C(20)-C(19)-C(22)	119.5(8)
C(18)-C(19)-C(22)	122.0(6)
C(21)-C(20)-C(19)	121.1(11)
C(21)-C(20)-H(20)	119.5
C(19)-C(20)-H(20)	119.5
C(20)-C(21)-C(16)	120.5(12)
C(20)-C(21)-H(21)	119.8
C(16)-C(21)-H(21)	119.8
C(21')-C(16')-C(17')	118.6(9)
C(21')-C(16')-C(8)	120.6(8)
C(17')-C(16')-C(8)	120.5(8)
C(18')-C(17')-C(16')	119.6(7)
C(18')-C(17')-H(17')	120.2
C(16')-C(17')-H(17')	120.2
C(19')-C(18')-C(17')	123.0(7)
C(19')-C(18')-H(18')	118.5
C(17')-C(18')-H(18')	118.5
C(18')-C(19')-C(20')	116.7(8)
C(18')-C(19')-C(22')	122.3(7)
C(20')-C(19')-C(22')	121.0(8)
C(19')-C(20')-C(21')	121.7(12)
C(19')-C(20')-H(20')	119.1
C(21')-C(20')-H(20')	119.1
C(16')-C(21')-C(20')	120.4(10)
C(16')-C(21')-H(21')	119.8
C(20')-C(21')-H(21')	119.8
C(19')-C(22')-H(22D)	109.5
C(19')-C(22')-H(22E)	109.5
H(22D)-C(22')-H(22E)	109.5
C(19')-C(22')-H(22F)	109.5
H(22D)-C(22')-H(22F)	109.5
H(22E)-C(22')-H(22F)	109.5
N(4)-C(23)-C(24)	120.8(3)
N(4)-C(23)-C(28)	114.9(3)
C(24)-C(23)-C(28)	124.3(3)
C(25)-C(24)-C(23)	120.0(4)
C(25)-C(24)-H(24)	120.0
C(23)-C(24)-H(24)	120.0
C(26)-C(25)-C(24)	118.9(4)
C(26)-C(25)-H(25)	120.5
C(24)-C(25)-H(25)	120.5
C(27)-C(26)-C(25)	119.7(4)
C(27)-C(26)-H(26)	120.2
C(25)-C(26)-H(26)	120.2
N(4)-C(27)-C(26)	122.5(4)
N(4)-C(27)-H(27)	118.8
C(26)-C(27)-H(27)	118.8
N(5)-C(28)-C(29)	121.5(3)
N(5)-C(28)-C(23)	114.4(3)
C(29)-C(28)-C(23)	124.1(3)
C(30)-C(29)-C(28)	119.2(4)
C(30)-C(29)-H(29)	120.4
C(28)-C(29)-H(29)	120.4
C(31)-C(30)-C(29)	119.1(4)
C(31)-C(30)-H(30)	120.4
C(29)-C(30)-H(30)	120.4
C(30)-C(31)-C(32)	119.6(4)

C(30)-C(31)-H(31)	120.2
C(32)-C(31)-H(31)	120.2
N(5)-C(32)-C(31)	122.4(4)
N(5)-C(32)-H(32)	118.8
C(31)-C(32)-H(32)	118.8
F(3)-P-F(6)	91.13(14)
F(3)-P-F(2)	90.49(14)
F(6)-P-F(2)	90.54(12)
F(3)-P-F(4)	178.54(14)
F(6)-P-F(4)	90.32(13)
F(2)-P-F(4)	89.67(13)
F(3)-P-F(5)	91.18(16)
F(6)-P-F(5)	90.43(14)
F(2)-P-F(5)	178.04(17)
F(4)-P-F(5)	88.63(15)
F(3)-P-F(1)	89.12(14)
F(6)-P-F(1)	179.73(17)
F(2)-P-F(1)	89.56(12)
F(4)-P-F(1)	89.43(13)
F(5)-P-F(1)	89.46(13)

Symmetry transformations used to generate equivalent atoms:

Table A2.60. Anisotropic displacement parameters ($Å^2$ x 10^3) for [Ru(ttp)(bpy)Cl](PF_6), **3.13.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Ru	12(1)	14(1)	13(1)	-1(1)	0(1)	2(1)
N(1)	14(1)	10(2)	11(2)	-1(1)	-1(1)	2(1)
N(2)	15(1)	24(2)	14(2)	-5(1)	2(1)	-1(1)
N(3)	12(2)	37(2)	16(2)	-6(1)	1(1)	6(1)
N(4)	15(2)	17(2)	15(2)	-5(1)	1(1)	-2(1)
N(5)	24(2)	13(2)	16(2)	0(1)	5(1)	1(1)
C(1)	17(2)	16(2)	12(2)	-2(2)	-2(1)	-3(1)
C(2)	12(2)	20(2)	19(2)	4(2)	0(1)	-2(1)
C(3)	19(2)	18(2)	17(2)	6(2)	6(1)	5(1)
C(4)	21(2)	12(2)	19(2)	0(2)	7(1)	2(1)
C(5)	17(2)	13(2)	11(2)	2(1)	1(1)	0(1)
C(6)	17(2)	20(2)	12(2)	-3(2)	2(1)	-3(1)
C(7)	25(2)	21(2)	23(2)	-9(2)	10(2)	-7(2)
C(8)	30(2)	46(3)	19(2)	-18(2)	12(2)	-22(2)
C(9)	18(2)	58(3)	23(2)	-22(2)	9(2)	-19(2)
C(10)	17(2)	38(2)	15(2)	-7(2)	5(1)	-2(2)
C(11)	15(2)	52(3)	16(2)	-14(2)	2(1)	-3(2)
C(12)	17(2)	87(4)	28(3)	-31(2)	0(2)	-2(2)
C(13)	17(2)	122(5)	39(3)	-48(3)	-1(2)	13(2)
C(14)	24(2)	88(4)	42(3)	-45(3)	-7(2)	26(2)
C(15)	27(2)	49(3)	23(2)	-16(2)	-2(2)	12(2)
C(16)	23(6)	16(7)	14(6)	1(5)	1(4)	-4(4)
C(17)	15(4)	19(4)	26(5)	-7(3)	7(3)	-4(3)
C(18)	32(5)	9(4)	22(5)	-8(3)	7(3)	1(3)
C(19)	23(4)	20(5)	15(4)	-1(4)	7(3)	-5(3)
C(20)	12(7)	16(9)	26(9)	-2(7)	-9(6)	-11(6)
C(21)	27(6)	12(6)	12(9)	-1(5)	-1(5)	8(5)
C(22)	38(5)	19(5)	45(6)	-12(4)	11(4)	-13(4)
C(16')	18(5)	13(6)	16(6)	1(4)	3(4)	-3(4)
C(17')	12(4)	17(4)	31(5)	-6(3)	4(3)	1(3)
C(18')	24(4)	21(4)	29(5)	-12(3)	4(3)	-1(3)
C(19')	28(4)	19(5)	26(5)	-11(4)	4(4)	-5(3)
C(20')	18(6)	6(6)	36(8)	1(5)	8(4)	0(5)
C(21')	24(5)	7(5)	10(8)	-6(4)	-3(4)	0(3)
C(22')	38(5)	32(5)	56(6)	-23(4)	20(4)	-13(4)
C(23)	20(2)	18(2)	16(2)	-3(2)	2(1)	3(2)
C(24)	33(2)	27(2)	15(2)	-3(2)	-4(2)	4(2)
C(25)	82(4)	25(3)	19(2)	3(2)	-6(2)	14(2)
C(26)	112(4)	14(2)	23(3)	3(2)	7(2)	-2(2)
C(27)	55(3)	16(2)	18(2)	-2(2)	5(2)	-11(2)
C(28)	13(2)	19(2)	20(2)	-5(2)	2(1)	-2(1)
C(29)	25(2)	27(2)	21(2)	-4(2)	4(2)	-5(2)
C(30)	59(3)	23(3)	27(3)	-15(2)	17(2)	-15(2)
C(31)	84(4)	12(2)	38(3)	-5(2)	27(2)	-10(2)
C(32)	53(3)	13(2)	24(2)	2(2)	12(2)	4(2)
Cl	16(1)	25(1)	18(1)	4(1)	2(1)	5(1)
P	40(1)	17(1)	29(1)	1(1)	19(1)	-2(1)
F(1)	71(2)	22(1)	31(1)	3(1)	32(1)	1(1)
F(2)	52(2)	18(1)	34(1)	5(1)	17(1)	-3(1)
F(3)	36(2)	79(2)	54(2)	-1(2)	18(1)	8(1)
F(4)	58(2)	31(1)	41(2)	6(1)	19(1)	21(1)
F(5)	97(2)	30(2)	68(2)	-13(1)	48(2)	-31(1)
F(6)	47(2)	47(2)	32(1)	-12(1)	17(1)	-10(1)

Table A2.61. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for [Ru(ttp)(bpy)Cl](PF$_6$), **3.13.**

	x	y	z	U(eq)
H(1)	9929	10699	7005	19
H(2)	11292	10066	6502	21
H(3)	10964	8952	5715	21
H(4)	9273	8504	5459	21
H(7)	7537	8152	5272	27
H(9)	4714	8818	5796	39
H(12)	3854	9792	6262	53
H(13)	3137	10775	6961	72
H(14)	4235	11671	7758	63
H(15)	6008	11574	7802	41
H(17)	7044	7031	5455	23
H(18)	6334	5931	5102	25
H(20)	3460	6757	4441	23
H(21)	4152	7871	4808	21
H(22A)	4138	5355	5286	50
H(22B)	4849	5177	4384	50
H(22C)	3728	5523	4005	50
H(17')	6474	7527	3901	24
H(18')	5556	6564	3109	30
H(20')	3398	6848	5033	24
H(21')	4279	7832	5814	17
H(22D)	3171	5891	3799	60
H(22E)	4218	5565	3510	60
H(22F)	3524	6074	2646	60
H(24)	9064	10526	10988	31
H(25)	8929	9336	11353	53
H(26)	8203	8592	9920	60
H(27)	7582	9042	8187	36
H(29)	9096	11639	10521	29
H(30)	9030	12793	9872	42
H(31)	8428	13008	7999	51
H(32)	7878	12088	6815	35

Table A2.62. Hydrogen bonds for [Ru(ttp)(bpy)Cl](PF$_6$), **3.13** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

Table A2.63. Crystal data and structure refinement for $[Ru(ttp)(bpy)(bpe)](PF_6)_2.MeOH$, **3.14.**

Identification code	**3.14**
Empirical formula	C45 H39 F12 N7 O P2 Ru
Formula weight	1084.84
Temperature	93(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, *p* 21/*c*
Unit cell dimensions	a = 10.7421(10) Å alpha = 90°. b = 21.633(2) Å beta = 99.463(2)°. c = 19.3655(14) Å gamma = 90°.
Volume	4438.9(7) $Å^3$
Z, Calculated density	4, 1.623 Mg/m^3
Absorption coefficient	0.522 mm^{-1}
F(000)	2192
Crystal size	0.59 x 0.27 x 0.21 mm
Theta range for data collection	1.88 to 26.41°.
Limiting indices	−13<=h<=13, −25<=k<=27, −24<=l<=24
Reflections collected / unique	38264 / 9001 [R(int) = 0.0337]
Completeness to theta = 26.41	98.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.8983 and 0.7483
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	9001 / 1119 / 807
Goodness-of-fit on F^2	1.027
Final R indices [I>2sigma(I)]	R1 = 0.0397, wR2 = 0.0933
R indices (all data)	R1 = 0.0541, wR2 = 0.1003
Largest diff. peak and hole	0.744 and −0.726 $eÅ^{-3}$

Table A2.64. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for [Ru(ttp)(bpy)(bpe)]$(PF_6)_2$.MeOH, **3.14.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ru	7151(1)	6981(1)	7012(1)	21(1)
N(1)	5402(5)	7405(2)	6826(3)	25(1)
N(2)	7536(5)	7754(2)	7525(3)	21(1)
N(3)	9064(5)	6871(2)	7354(3)	24(1)
N(4)	7567(5)	7279(3)	6068(3)	25(1)
N(5)	6769(5)	6193(2)	6399(3)	25(1)
C(1)	4323(6)	7201(3)	6434(4)	30(2)
C(2)	3190(7)	7506(3)	6401(4)	36(2)
C(3)	3134(7)	8038(3)	6795(4)	36(2)
C(4)	4238(6)	8261(3)	7186(4)	30(1)
C(5)	5356(6)	7949(3)	7182(3)	24(1)
C(6)	6597(6)	8162(3)	7564(3)	22(1)
C(7)	6854(6)	8721(3)	7901(3)	23(1)
C(8)	8093(6)	8866(3)	8222(3)	22(1)
C(9)	9030(6)	8419(3)	8191(3)	22(1)
C(10)	8739(6)	7872(3)	7836(3)	21(1)
C(11)	9618(6)	7367(3)	7726(3)	24(1)
C(12)	10900(7)	7380(3)	7970(4)	32(2)
C(13)	11644(7)	6883(4)	7837(4)	40(2)
C(14)	11086(7)	6387(4)	7461(4)	41(2)
C(15)	9793(7)	6396(3)	7229(4)	32(2)
C(16)	8401(6)	9469(3)	8572(3)	23(1)
C(17)	7452(6)	9878(3)	8698(3)	25(1)
C(18)	7743(6)	10443(3)	9023(3)	25(1)
C(19)	8987(6)	10623(3)	9242(3)	28(1)
C(20)	9925(7)	10222(3)	9105(4)	35(2)
C(21)	9648(6)	9661(3)	8773(4)	31(2)
C(22)	9312(7)	11233(3)	9598(4)	36(2)
C(23)	8033(7)	7840(3)	5946(3)	30(2)
C(24)	8363(7)	8003(3)	5312(4)	37(2)
C(25)	8174(8)	7582(4)	4769(4)	39(2)
C(26)	7700(7)	7009(3)	4881(3)	33(2)
C(27)	7414(6)	6856(3)	5537(3)	26(1)
C(28)	6969(6)	6251(3)	5724(3)	26(1)
C(29)	6785(6)	5752(3)	5265(4)	31(2)
C(30)	6419(6)	5189(3)	5498(4)	34(2)
C(31)	6199(6)	5134(3)	6177(4)	33(2)
C(32)	6374(6)	5650(3)	6609(4)	30(2)
N(6)	6810(50)	6560(30)	7940(30)	20(6)
C(33)	5900(30)	6730(20)	8300(20)	27(6)
C(34)	5700(20)	6482(12)	8923(12)	26(4)
C(35)	6518(18)	6037(8)	9249(10)	23(4)
C(36)	7504(18)	5868(9)	8906(10)	23(4)
C(37)	7630(30)	6124(14)	8273(17)	25(5)
C(38)	6415(15)	5749(6)	9921(6)	29(3)
C(39)	5460(12)	5814(5)	10261(7)	32(3)
C(40)	5377(18)	5508(7)	10935(6)	27(3)
C(41)	6353(15)	5181(9)	11319(8)	32(3)
C(42)	6180(20)	4918(9)	11949(10)	36(4)
N(7)	5120(50)	4954(18)	12210(20)	27(5)
C(43)	4160(30)	5260(20)	11830(19)	27(5)
C(44)	4241(18)	5540(8)	11189(8)	28(3)

N(6')	6670(110)	6630(60)	7980(60)	26(12)
C(33')	7350(60)	6190(30)	8320(40)	28(9)
C(34')	7090(40)	5940(20)	8950(20)	27(7)
C(35')	6080(30)	6140(20)	9220(20)	24(7)
C(36')	5320(40)	6580(30)	8850(30)	24(7)
C(37')	5610(60)	6790(40)	8220(40)	19(8)
C(38')	5720(30)	5903(12)	9882(14)	31(6)
C(39')	6320(20)	5494(10)	10333(11)	28(6)
C(40')	5880(30)	5274(15)	10979(15)	24(6)
C(41')	4750(30)	5466(16)	11137(18)	27(6)
C(42')	4360(80)	5230(50)	11740(40)	37(10)
N(7')	5040(110)	4820(40)	12200(50)	41(14)
C(43')	6150(40)	4660(19)	12000(20)	41(9)
C(44')	6600(30)	4868(15)	11425(13)	31(6)
P(1)	3751(2)	8226(1)	9295(1)	28(1)
F(11)	3798(4)	8693(2)	9945(2)	38(1)
F(12)	2700(4)	8647(2)	8839(2)	44(1)
F(13)	2650(4)	7839(2)	9569(2)	40(1)
F(14)	3707(4)	7758(2)	8650(2)	40(1)
F(15)	4780(4)	7803(2)	9756(2)	42(1)
F(16)	4839(4)	8616(2)	9019(2)	44(1)
P(2)	9773(5)	4520(3)	8325(3)	30(1)
F(21)	8678(11)	4414(5)	8771(5)	41(3)
F(22)	9930(8)	5212(4)	8567(6)	58(3)
F(23)	10789(11)	4319(6)	8967(7)	70(4)
F(24)	10850(7)	4646(7)	7867(5)	66(4)
F(25)	8731(8)	4769(8)	7685(4)	83(5)
F(26)	9591(11)	3853(5)	8015(9)	113(7)
P(2')	9772(16)	4338(8)	8464(8)	49(4)
F(21')	8520(30)	4464(16)	8818(16)	103(17)
F(22')	9810(30)	4957(11)	8291(17)	160(20)
F(23')	10640(20)	4454(13)	9208(10)	68(9)
F(24')	11041(14)	4210(11)	8144(11)	61(7)
F(25')	8926(16)	4295(11)	7710(8)	55(6)
F(26')	9665(15)	3694(7)	8594(12)	59(6)
C	1449(18)	6304(8)	9636(8)	132(7)
O	2731(10)	6489(6)	9644(9)	140(5)

Table A2.65. Selected bond lengths [Å] and angles [°] for [Ru(ttp)(bpy)(bpe)]$(PF_6)_2$.MeOH, **3.14.**

Ru–N(2)	1.955(5)
Ru–N(4)	2.056(5)
Ru–N(3)	2.067(5)
Ru–N(1)	2.068(5)
Ru–N(5)	2.078(5)
Ru–N(6)	2.10(4)
N(2)–Ru–N(4)	97.2(2)
N(2)–Ru–N(3)	79.6(2)
N(4)–Ru–N(3)	88.3(2)
N(2)–Ru–N(1)	79.6(2)
N(4)–Ru–N(1)	91.7(2)
N(3)–Ru–N(1)	159.0(2)
N(2)–Ru–N(5)	175.7(2)
N(4)–Ru–N(5)	78.5(2)
N(3)–Ru–N(5)	100.4(2)
N(1)–Ru–N(5)	100.1(2)
N(2)–Ru–N(6)	89.1(18)
N(4)–Ru–N(6)	172.5(15)
N(3)–Ru–N(6)	88.9(15)
N(1)–Ru–N(6)	93.3(13)
N(5)–Ru–N(6)	95.2(18)

Symmetry transformations used to generate equivalent atoms:

Table A2.66. Bond lengths [Å] and angles [°] for $[Ru(ttp)(bpy)(bpe)](PF_6)_2.MeOH$, **3.14.**

Ru–N(2)	1.955(5)
Ru–N(4)	2.056(5)
Ru–N(3)	2.067(5)
Ru–N(1)	2.068(5)
Ru–N(5)	2.078(5)
Ru–N(6)	2.10(4)
Ru–N(6')	2.16(10)
N(1)–C(1)	1.350(8)
N(1)–C(5)	1.370(8)
N(2)–C(6)	1.351(8)
N(2)–C(10)	1.357(8)
N(3)–C(15)	1.338(8)
N(3)–C(11)	1.371(8)
N(4)–C(23)	1.348(9)
N(4)–C(27)	1.365(8)
N(5)–C(32)	1.336(9)
N(5)–C(28)	1.366(8)
C(1)–C(2)	1.376(10)
C(1)–H(1)	0.9500
C(2)–C(3)	1.387(10)
C(2)–H(2)	0.9500
C(3)–C(4)	1.385(10)
C(3)–H(3)	0.9500
C(4)–C(5)	1.379(9)
C(4)–H(4)	0.9500
C(5)–C(6)	1.486(9)
C(6)–C(7)	1.382(8)
C(7)–C(8)	1.407(9)
C(7)–H(7)	0.9500
C(8)–C(9)	1.405(9)
C(8)–C(16)	1.483(9)
C(9)–C(10)	1.378(8)
C(9)–H(9)	0.9500
C(10)–C(11)	1.482(9)
C(11)–C(12)	1.382(9)
C(12)–C(13)	1.390(10)
C(12)–H(12)	0.9500
C(13)–C(14)	1.377(10)
C(13)–H(13)	0.9500
C(14)–C(15)	1.387(10)
C(14)–H(14)	0.9500
C(15)–H(15)	0.9500
C(16)–C(21)	1.395(9)
C(16)–C(17)	1.401(9)
C(17)–C(18)	1.386(9)
C(17)–H(17)	0.9500
C(18)–C(19)	1.390(9)
C(18)–H(18)	0.9500
C(19)–C(20)	1.389(10)
C(19)–C(22)	1.504(9)
C(20)–C(21)	1.382(9)
C(20)–H(20)	0.9500
C(21)–H(21)	0.9500
C(22)–H(22A)	0.9800
C(22)–H(22B)	0.9800
C(22)–H(22C)	0.9800

C(23)-C(24)	1.379(10)
C(23)-H(23)	0.9500
C(24)-C(25)	1.381(10)
C(24)-H(24)	0.9500
C(25)-C(26)	1.370(11)
C(25)-H(25)	0.9500
C(26)-C(27)	1.395(9)
C(26)-H(26)	0.9500
C(27)-C(28)	1.461(10)
C(28)-C(29)	1.392(9)
C(29)-C(30)	1.378(10)
C(29)-H(29)	0.9500
C(30)-C(31)	1.380(10)
C(30)-H(30)	0.9500
C(31)-C(32)	1.387(9)
C(31)-H(31)	0.9500
C(32)-H(32)	0.9500
N(6)-C(33)	1.34(4)
N(6)-C(37)	1.38(4)
C(33)-C(34)	1.38(4)
C(33)-H(33)	0.9500
C(34)-C(35)	1.38(2)
C(34)-H(34)	0.9500
C(35)-C(36)	1.388(18)
C(35)-C(38)	1.46(2)
C(36)-C(37)	1.37(3)
C(36)-H(36)	0.9500
C(37)-H(37)	0.9500
C(38)-C(39)	1.31(2)
C(38)-H(38)	0.9500
C(39)-C(40)	1.481(18)
C(39)-H(39)	0.9500
C(40)-C(41)	1.38(2)
C(40)-C(44)	1.391(18)
C(41)-C(42)	1.39(2)
C(41)-H(41)	0.9500
C(42)-N(7)	1.33(5)
C(42)-H(42)	0.9500
N(7)-C(43)	1.34(6)
C(43)-C(44)	1.39(3)
C(43)-H(43)	0.9500
C(44)-H(44)	0.9500
N(6')-C(37')	1.35(9)
N(6')-C(33')	1.30(10)
C(33')-C(34')	1.41(7)
C(33')-H(33')	0.9500
C(34')-C(35')	1.35(4)
C(34')-H(34')	0.9500
C(35')-C(36')	1.37(4)
C(35')-C(38')	1.49(5)
C(36')-C(37')	1.39(8)
C(36')-H(36')	0.9500
C(37')-H(37')	0.9500
C(38')-C(39')	1.34(4)
C(38')-H(38')	0.9500
C(39')-C(40')	1.49(3)
C(39')-H(39')	0.9500
C(40')-C(41')	1.37(4)
C(40')-C(44')	1.38(3)
C(41')-C(42')	1.39(7)
C(41')-H(41')	0.9500

C(42')-N(7')	1.38(12)
C(42')-H(42')	0.9500
N(7')-C(43')	1.36(12)
C(43')-C(44')	1.37(4)
C(43')-H(43')	0.9500
C(44')-H(44')	0.9500
P(1)-F(15)	1.591(4)
P(1)-F(12)	1.598(4)
P(1)-F(14)	1.603(4)
P(1)-F(16)	1.603(4)
P(1)-F(13)	1.608(4)
P(1)-F(11)	1.608(4)
P(2)-F(22)	1.570(9)
P(2)-F(21)	1.586(10)
P(2)-F(26)	1.562(10)
P(2)-F(24)	1.593(9)
P(2)-F(23)	1.576(10)
P(2)-F(25)	1.619(10)
P(2')-F(23')	1.60(2)
P(2')-F(25')	1.591(19)
P(2')-F(24')	1.612(19)
P(2')-F(26')	1.423(18)
P(2')-F(21')	1.63(2)
P(2')-F(22')	1.381(18)
C-O	1.432(18)
C-H(0A)	0.9800
C-H(0B)	0.9800
C-H(0C)	0.9800
O-H(0)	0.8400

N(2)-Ru-N(4)	97.2(2)
N(2)-Ru-N(3)	79.6(2)
N(4)-Ru-N(3)	88.3(2)
N(2)-Ru-N(1)	79.6(2)
N(4)-Ru-N(1)	91.7(2)
N(3)-Ru-N(1)	159.0(2)
N(2)-Ru-N(5)	175.7(2)
N(4)-Ru-N(5)	78.5(2)
N(3)-Ru-N(5)	100.4(2)
N(1)-Ru-N(5)	100.1(2)
N(2)-Ru-N(6)	89.1(18)
N(4)-Ru-N(6)	172.5(15)
N(3)-Ru-N(6)	88.9(15)
N(1)-Ru-N(6)	93.3(13)
N(5)-Ru-N(6)	95.2(18)
N(2)-Ru-N(6')	86(4)
N(4)-Ru-N(6')	177(4)
N(3)-Ru-N(6')	93(3)
N(1)-Ru-N(6')	88(3)
N(5)-Ru-N(6')	99(4)
N(6)-Ru-N(6')	6(4)
C(1)-N(1)-C(5)	117.7(6)
C(1)-N(1)-Ru	128.7(4)
C(5)-N(1)-Ru	113.6(4)
C(6)-N(2)-C(10)	121.3(5)
C(6)-N(2)-Ru	119.3(4)
C(10)-N(2)-Ru	119.5(4)
C(15)-N(3)-C(11)	118.7(6)
C(15)-N(3)-Ru	127.7(5)
C(11)-N(3)-Ru	113.6(4)
C(23)-N(4)-C(27)	118.2(6)

C(23)-N(4)-Ru	125.4(4)
C(27)-N(4)-Ru	116.3(4)
C(32)-N(5)-C(28)	118.6(6)
C(32)-N(5)-Ru	126.2(4)
C(28)-N(5)-Ru	115.2(4)
N(1)-C(1)-C(2)	122.7(6)
N(1)-C(1)-H(1)	118.6
C(2)-C(1)-H(1)	118.6
C(1)-C(2)-C(3)	119.3(6)
C(1)-C(2)-H(2)	120.3
C(3)-C(2)-H(2)	120.3
C(2)-C(3)-C(4)	118.6(7)
C(2)-C(3)-H(3)	120.7
C(4)-C(3)-H(3)	120.7
C(5)-C(4)-C(3)	119.7(6)
C(5)-C(4)-H(4)	120.2
C(3)-C(4)-H(4)	120.2
N(1)-C(5)-C(4)	121.8(6)
N(1)-C(5)-C(6)	114.4(5)
C(4)-C(5)-C(6)	123.9(6)
N(2)-C(6)-C(7)	120.3(6)
N(2)-C(6)-C(5)	112.8(5)
C(7)-C(6)-C(5)	126.8(6)
C(6)-C(7)-C(8)	120.3(6)
C(6)-C(7)-H(7)	119.9
C(8)-C(7)-H(7)	119.9
C(9)-C(8)-C(7)	117.4(6)
C(9)-C(8)-C(16)	121.2(6)
C(7)-C(8)-C(16)	121.4(6)
C(10)-C(9)-C(8)	120.5(6)
C(10)-C(9)-H(9)	119.8
C(8)-C(9)-H(9)	119.8
N(2)-C(10)-C(9)	120.2(6)
N(2)-C(10)-C(11)	112.4(5)
C(9)-C(10)-C(11)	127.4(6)
N(3)-C(11)-C(12)	121.2(6)
N(3)-C(11)-C(10)	114.9(5)
C(12)-C(11)-C(10)	123.9(6)
C(11)-C(12)-C(13)	119.4(6)
C(11)-C(12)-H(12)	120.3
C(13)-C(12)-H(12)	120.3
C(14)-C(13)-C(12)	119.2(7)
C(14)-C(13)-H(13)	120.4
C(12)-C(13)-H(13)	120.4
C(13)-C(14)-C(15)	119.1(7)
C(13)-C(14)-H(14)	120.5
C(15)-C(14)-H(14)	120.5
N(3)-C(15)-C(14)	122.4(6)
N(3)-C(15)-H(15)	118.8
C(14)-C(15)-H(15)	118.8
C(21)-C(16)-C(17)	117.2(6)
C(21)-C(16)-C(8)	121.4(6)
C(17)-C(16)-C(8)	121.4(6)
C(18)-C(17)-C(16)	121.2(6)
C(18)-C(17)-H(17)	119.4
C(16)-C(17)-H(17)	119.4
C(17)-C(18)-C(19)	121.4(6)
C(17)-C(18)-H(18)	119.3
C(19)-C(18)-H(18)	119.3
C(18)-C(19)-C(20)	117.2(6)
C(18)-C(19)-C(22)	121.7(6)

C(20)-C(19)-C(22)	121.1(6)
C(21)-C(20)-C(19)	122.0(7)
C(21)-C(20)-H(20)	119.0
C(19)-C(20)-H(20)	119.0
C(20)-C(21)-C(16)	120.9(6)
C(20)-C(21)-H(21)	119.5
C(16)-C(21)-H(21)	119.5
C(19)-C(22)-H(22A)	109.5
C(19)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(19)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
N(4)-C(23)-C(24)	123.0(6)
N(4)-C(23)-H(23)	118.5
C(24)-C(23)-H(23)	118.5
C(23)-C(24)-C(25)	118.7(7)
C(23)-C(24)-H(24)	120.7
C(25)-C(24)-H(24)	120.7
C(26)-C(25)-C(24)	119.3(7)
C(26)-C(25)-H(25)	120.3
C(24)-C(25)-H(25)	120.3
C(25)-C(26)-C(27)	120.0(6)
C(25)-C(26)-H(26)	120.0
C(27)-C(26)-H(26)	120.0
N(4)-C(27)-C(26)	120.7(6)
N(4)-C(27)-C(28)	114.7(6)
C(26)-C(27)-C(28)	124.6(6)
N(5)-C(28)-C(29)	120.9(6)
N(5)-C(28)-C(27)	115.3(5)
C(29)-C(28)-C(27)	123.8(6)
C(30)-C(29)-C(28)	119.6(7)
C(30)-C(29)-H(29)	120.2
C(28)-C(29)-H(29)	120.2
C(29)-C(30)-C(31)	119.4(6)
C(29)-C(30)-H(30)	120.3
C(31)-C(30)-H(30)	120.3
C(30)-C(31)-C(32)	118.6(7)
C(30)-C(31)-H(31)	120.7
C(32)-C(31)-H(31)	120.7
N(5)-C(32)-C(31)	122.9(7)
N(5)-C(32)-H(32)	118.6
C(31)-C(32)-H(32)	118.6
C(33)-N(6)-C(37)	115(3)
C(33)-N(6)-Ru	125(3)
C(37)-N(6)-Ru	120(3)
N(6)-C(33)-C(34)	125(3)
N(6)-C(33)-H(33)	117.4
C(34)-C(33)-H(33)	117.4
C(33)-C(34)-C(35)	119.9(19)
C(33)-C(34)-H(34)	120.1
C(35)-C(34)-H(34)	120.1
C(36)-C(35)-C(34)	116.3(15)
C(36)-C(35)-C(38)	119.2(14)
C(34)-C(35)-C(38)	124.5(16)
C(37)-C(36)-C(35)	121.0(16)
C(37)-C(36)-H(36)	119.5
C(35)-C(36)-H(36)	119.5
N(6)-C(37)-C(36)	123(2)
N(6)-C(37)-H(37)	118.5

C(36)-C(37)-H(37)	118.5
C(39)-C(38)-C(35)	125.4(14)
C(39)-C(38)-H(38)	117.3
C(35)-C(38)-H(38)	117.3
C(38)-C(39)-C(40)	123.9(13)
C(38)-C(39)-H(39)	118.0
C(40)-C(39)-H(39)	118.0
C(41)-C(40)-C(44)	118.0(12)
C(41)-C(40)-C(39)	123.7(16)
C(44)-C(40)-C(39)	118.2(15)
C(40)-C(41)-C(42)	119.1(14)
C(40)-C(41)-H(41)	120.4
C(42)-C(41)-H(41)	120.4
N(7)-C(42)-C(41)	124(2)
N(7)-C(42)-H(42)	117.9
C(41)-C(42)-H(42)	117.9
C(43)-N(7)-C(42)	116(3)
N(7)-C(43)-C(44)	124(3)
N(7)-C(43)-H(43)	118.2
C(44)-C(43)-H(43)	118.2
C(43)-C(44)-C(40)	118.5(17)
C(43)-C(44)-H(44)	120.7
C(40)-C(44)-H(44)	120.7
C(37')-N(6')-C(33')	117(8)
C(37')-N(6')-Ru	123(7)
C(33')-N(6')-Ru	120(6)
C(34')-C(33')-N(6')	123(5)
C(34')-C(33')-H(33')	118.5
N(6')-C(33')-H(33')	118.5
C(35')-C(34')-C(33')	120(4)
C(35')-C(34')-H(34')	120.2
C(33')-C(34')-H(34')	120.2
C(34')-C(35')-C(36')	118(3)
C(34')-C(35')-C(38')	124(3)
C(36')-C(35')-C(38')	118(3)
C(37')-C(36')-C(35')	120(4)
C(37')-C(36')-H(36')	120.1
C(35')-C(36')-H(36')	120.1
N(6')-C(37')-C(36')	122(6)
N(6')-C(37')-H(37')	118.8
C(36')-C(37')-H(37')	118.8
C(39')-C(38')-C(35')	128(3)
C(39')-C(38')-H(38')	115.8
C(35')-C(38')-H(38')	115.8
C(38')-C(39')-C(40')	125(3)
C(38')-C(39')-H(39')	117.6
C(40')-C(39')-H(39')	117.6
C(41')-C(40')-C(44')	119(3)
C(41')-C(40')-C(39')	121(3)
C(44')-C(40')-C(39')	120(3)
C(40')-C(41')-C(42')	118(4)
C(40')-C(41')-H(41')	121.0
C(42')-C(41')-H(41')	120.9
N(7')-C(42')-C(41')	126(7)
N(7')-C(42')-H(42')	117.1
C(41')-C(42')-H(42')	117.1
C(43')-N(7')-C(42')	112(7)
N(7')-C(43')-C(44')	127(5)
N(7')-C(43')-H(43')	116.7
C(44')-C(43')-H(43')	116.7

C(43')-C(44')-C(40')	119(3)
C(43')-C(44')-H(44')	120.7
C(40')-C(44')-H(44')	120.7
F(15)-P(1)-F(12)	179.1(3)
F(15)-P(1)-F(14)	90.1(2)
F(12)-P(1)-F(14)	90.0(2)
F(15)-P(1)-F(16)	90.7(3)
F(12)-P(1)-F(16)	90.2(3)
F(14)-P(1)-F(16)	90.1(2)
F(15)-P(1)-F(13)	89.8(3)
F(12)-P(1)-F(13)	89.3(3)
F(14)-P(1)-F(13)	90.0(2)
F(16)-P(1)-F(13)	179.5(3)
F(15)-P(1)-F(11)	89.6(2)
F(12)-P(1)-F(11)	90.2(2)
F(14)-P(1)-F(11)	179.7(3)
F(16)-P(1)-F(11)	90.0(2)
F(13)-P(1)-F(11)	89.9(2)
F(22)-P(2)-F(21)	91.6(6)
F(22)-P(2)-F(26)	174.9(8)
F(21)-P(2)-F(26)	91.1(6)
F(22)-P(2)-F(24)	87.1(6)
F(21)-P(2)-F(24)	178.1(7)
F(26)-P(2)-F(24)	90.1(6)
F(22)-P(2)-F(23)	90.0(7)
F(21)-P(2)-F(23)	90.5(6)
F(26)-P(2)-F(23)	94.3(8)
F(24)-P(2)-F(23)	90.8(6)
F(22)-P(2)-F(25)	86.4(6)
F(21)-P(2)-F(25)	89.0(6)
F(26)-P(2)-F(25)	89.3(7)
F(24)-P(2)-F(25)	89.5(5)
F(23)-P(2)-F(25)	176.4(8)
F(23')-P(2')-F(25')	174.3(16)
F(23')-P(2')-F(24')	88.2(12)
F(25')-P(2')-F(24')	91.6(11)
F(23')-P(2')-F(26')	92.7(13)
F(25')-P(2')-F(26')	93.1(13)
F(24')-P(2')-F(26')	89.9(12)
F(23')-P(2')-F(21')	89.8(13)
F(25')-P(2')-F(21')	90.5(14)
F(24')-P(2')-F(21')	177.9(16)
F(26')-P(2')-F(21')	89.6(14)
F(23')-P(2')-F(22')	91.8(15)
F(25')-P(2')-F(22')	82.4(17)
F(24')-P(2')-F(22')	90.9(14)
F(26')-P(2')-F(22')	175.5(19)
F(21')-P(2')-F(22')	89.8(15)
O-C-H(0A)	109.5
O-C-H(0B)	109.5
H(0A)-C-H(0B)	109.5
O-C-H(0C)	109.5
H(0A)-C-H(0C)	109.5
H(0B)-C-H(0C)	109.5
C-O-H(0)	109.5

Symmetry transformations used to generate equivalent atoms:

Table A2.67. Anisotropic displacement parameters ($Å^2$ x 10^3) for [Ru(ttp)(bpy)(bpe)]$(PF_6)_2$.MeOH, **3.14.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Ru	22(1)	21(1)	19(1)	-4(1)	0(1)	1(1)
N(1)	23(3)	25(3)	25(3)	-2(2)	1(2)	-1(2)
N(2)	22(3)	21(3)	19(2)	-1(2)	3(2)	1(2)
N(3)	24(3)	25(3)	23(3)	-3(2)	0(2)	3(2)
N(4)	22(3)	27(3)	23(3)	-1(2)	-1(2)	4(2)
N(5)	19(3)	27(3)	25(3)	-6(2)	-2(2)	3(2)
C(1)	26(4)	31(4)	30(3)	-6(3)	-2(3)	-2(3)
C(2)	25(4)	35(4)	45(4)	-6(3)	-5(3)	-3(3)
C(3)	22(3)	33(4)	52(4)	-3(3)	0(3)	4(3)
C(4)	25(3)	26(3)	37(4)	-3(3)	2(3)	1(3)
C(5)	23(3)	21(3)	26(3)	1(2)	2(2)	-1(3)
C(6)	22(3)	22(3)	21(3)	0(2)	2(2)	0(2)
C(7)	21(3)	22(3)	26(3)	-2(2)	4(2)	2(2)
C(8)	23(3)	22(3)	21(3)	1(2)	4(2)	0(2)
C(9)	21(3)	24(3)	21(3)	0(2)	1(2)	0(2)
C(10)	21(3)	22(3)	20(3)	1(2)	2(2)	1(2)
C(11)	25(3)	26(3)	22(3)	-3(2)	1(2)	2(3)
C(12)	28(4)	30(4)	34(4)	-8(3)	-2(3)	3(3)
C(13)	25(4)	44(5)	49(4)	-13(3)	-6(3)	9(3)
C(14)	34(4)	37(4)	49(5)	-14(3)	-3(3)	13(3)
C(15)	32(4)	29(4)	33(4)	-9(3)	-1(3)	5(3)
C(16)	25(3)	23(3)	22(3)	0(2)	4(2)	0(3)
C(17)	23(3)	26(3)	24(3)	-2(2)	2(3)	1(3)
C(18)	27(3)	24(3)	25(3)	-2(2)	5(3)	3(3)
C(19)	32(4)	22(3)	29(3)	-2(3)	4(3)	-1(3)
C(20)	23(4)	28(4)	52(4)	-8(3)	2(3)	-4(3)
C(21)	24(3)	22(3)	47(4)	-7(3)	4(3)	1(3)
C(22)	34(4)	28(4)	43(4)	-10(3)	4(3)	-2(3)
C(23)	35(4)	29(4)	25(3)	-2(3)	3(3)	3(3)
C(24)	48(4)	30(4)	32(4)	5(3)	6(3)	4(3)
C(25)	51(5)	40(4)	27(4)	7(3)	6(3)	10(4)
C(26)	38(4)	37(4)	21(3)	-7(3)	-2(3)	11(3)
C(27)	22(3)	32(4)	22(3)	-4(2)	-3(2)	10(3)
C(28)	18(3)	33(4)	24(3)	-6(3)	-4(2)	6(3)
C(29)	28(4)	37(4)	26(3)	-10(3)	-2(3)	5(3)
C(30)	25(4)	35(4)	38(4)	-16(3)	-3(3)	2(3)
C(31)	24(4)	30(4)	44(4)	-10(3)	1(3)	-3(3)
C(32)	27(4)	30(4)	31(4)	-5(3)	2(3)	-1(3)
N(6)	27(14)	14(9)	19(7)	-4(6)	2(8)	3(8)
C(33)	22(15)	27(9)	30(10)	3(6)	-2(10)	7(10)
C(34)	22(11)	29(11)	28(8)	-3(6)	5(9)	8(8)
C(35)	26(11)	17(8)	23(6)	-3(5)	-2(7)	5(7)
C(36)	28(10)	17(7)	25(6)	2(4)	3(7)	8(7)
C(37)	30(13)	17(8)	30(8)	0(6)	5(7)	9(7)
C(38)	36(8)	25(6)	26(6)	-1(4)	1(5)	0(5)
C(39)	39(7)	30(6)	26(6)	0(4)	3(5)	0(5)
C(40)	23(9)	30(7)	27(6)	-5(5)	2(5)	-5(6)
C(41)	24(7)	47(10)	25(7)	2(7)	0(5)	-3(7)
C(42)	31(7)	48(11)	28(7)	0(7)	-1(5)	2(9)
N(7)	29(9)	30(12)	24(7)	5(8)	4(6)	-4(9)
C(43)	35(12)	26(9)	21(9)	0(7)	4(7)	-1(8)
C(44)	30(9)	29(7)	26(6)	1(5)	4(7)	2(7)
N(6')	23(19)	20(30)	31(19)	-7(16)	5(13)	-1(16)

C(33')	13(19)	33(19)	38(15)	-23(13)	3(12)	4(13)
C(34')	18(18)	24(14)	39(14)	-3(11)	1(12)	15(13)
C(35')	15(17)	26(16)	27(11)	2(9)	-4(12)	14(13)
C(36')	15(19)	24(16)	31(13)	8(10)	0(14)	11(13)
C(37')	10(20)	23(19)	21(15)	-1(10)	-3(15)	4(14)
C(38')	35(15)	29(12)	27(12)	-1(9)	2(11)	1(11)
C(39')	30(12)	28(12)	25(10)	0(8)	2(8)	-3(9)
C(40')	19(14)	29(14)	22(11)	-4(9)	-3(10)	5(10)
C(41')	10(16)	33(14)	33(13)	-4(11)	-9(11)	0(13)
C(42')	30(20)	35(19)	40(30)	-16(17)	9(14)	-9(15)
N(7')	34(18)	50(40)	36(16)	-4(18)	-2(12)	0(20)
C(43')	39(14)	50(20)	33(13)	6(15)	3(10)	0(17)
C(44')	32(12)	31(14)	26(11)	7(10)	-8(9)	3(10)
P(1)	26(1)	29(1)	28(1)	-7(1)	3(1)	-1(1)
F(11)	43(2)	34(2)	38(2)	-13(2)	8(2)	-2(2)
F(12)	40(3)	39(2)	48(3)	2(2)	-7(2)	5(2)
F(13)	38(2)	35(2)	49(3)	-6(2)	14(2)	-7(2)
F(14)	37(2)	45(2)	36(2)	-19(2)	4(2)	-4(2)
F(15)	38(2)	41(2)	43(2)	-8(2)	-6(2)	10(2)
F(16)	40(3)	51(3)	42(2)	-9(2)	10(2)	-14(2)
P(2)	24(2)	33(3)	35(3)	-2(2)	9(2)	-2(2)
F(21)	28(5)	54(6)	43(6)	6(4)	14(4)	-2(4)
F(22)	51(5)	24(4)	99(7)	11(4)	11(5)	-8(4)
F(23)	29(5)	84(8)	92(9)	59(7)	-6(6)	1(5)
F(24)	36(4)	115(10)	52(5)	-12(5)	17(4)	-14(5)
F(25)	42(5)	160(14)	44(5)	22(6)	-4(4)	-2(7)
F(26)	90(8)	80(9)	189(18)	-91(11)	85(11)	-37(7)
P(2')	48(6)	51(9)	48(7)	11(5)	11(5)	-11(6)
F(21')	49(19)	160(40)	100(30)	20(20)	27(18)	30(20)
F(22')	150(30)	70(30)	210(40)	10(30)	-80(30)	-40(20)
F(23')	47(15)	102(19)	50(13)	-23(12)	-6(10)	-13(12)
F(24')	47(10)	76(16)	64(13)	-28(11)	19(9)	-7(10)
F(25')	53(11)	64(14)	44(9)	-1(9)	-6(8)	6(10)
F(26')	28(9)	57(11)	87(15)	-1(10)	-7(9)	-11(7)
C	167(17)	138(14)	89(10)	28(10)	15(11)	-72(13)
O	97(8)	115(9)	216(14)	36(9)	51(8)	14(7)

Table A2.68. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for [Ru(ttp)(bpy)(bpe)]$(PF_6)_2$.MeOH, **3.14.**

	x	y	z	U(eq)
H(1)	4347	6833	6170	36
H(2)	2454	7354	6112	43
H(3)	2354	8245	6796	44
H(4)	4225	8628	7455	36
H(7)	6193	9010	7917	28
H(9)	9871	8494	8415	27
H(12)	11270	7727	8227	38
H(13)	12527	6884	8004	49
H(14)	11579	6045	7360	49
H(15)	9411	6052	6973	39
H(17)	6592	9767	8557	30
H(18)	7079	10712	9097	30
H(20)	10784	10336	9243	42
H(21)	10316	9402	8680	38
H(22A)	9583	11525	9264	53
H(22B)	9996	11175	9994	53
H(22C)	8567	11399	9767	53
H(23)	8139	8135	6313	36
H(24)	8715	8399	5250	44
H(25)	8369	7688	4323	47
H(26)	7567	6715	4511	39
H(29)	6911	5799	4793	37
H(30)	6319	4841	5193	40
H(31)	5933	4752	6346	40
H(32)	6206	5615	7074	36
H(33)	5343	7053	8102	32
H(34)	5004	6614	9131	31
H(36)	8101	5570	9113	28
H(37)	8313	5994	8053	31
H(38)	7093	5493	10128	35
H(39)	4782	6074	10062	39
H(41)	7133	5136	11154	39
H(42)	6869	4698	12209	44
H(43)	3389	5291	12004	33
H(44)	3533	5747	10932	34
H(33')	8052	6035	8130	34
H(34')	7626	5629	9190	33
H(36')	4598	6728	9019	29
H(37')	5039	7069	7946	23
H(38')	4953	6064	9999	37
H(39')	7099	5333	10235	33
H(41')	4237	5752	10844	32
H(42')	3567	5363	11835	44
H(43')	6657	4373	12293	50
H(44')	7403	4734	11333	37
H(0A)	1379	5855	9577	198
H(0B)	905	6508	9246	198
H(0C)	1184	6423	10078	198
H(0)	2751	6861	9523	210

Table A2.69. Hydrogen bonds for [Ru(ttp)(bpy)(bpe)]$(PF_6)_2$.MeOH, **3.14** [Å and °].

D–H...A	d(D–H)	d(H...A)	d(D...A)	<(DHA)
O–H(0)...F(13)	0.84	2.12	2.926(13)	160.1

Symmetry transformations used to generate equivalent atoms:

Table A2.70. Crystal data and structure refinement for pzt, **1.71.**

Identification code	**1.71**
Empirical formula	C33 H27 N9 O
Formula weight	565.64
Temperature	191(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, *pbca*
Unit cell dimensions	a = 11.429(11) Å alpha = 90°. b = 8.715(7) Å beta = 90°. c = 55.97(5) Å gamma = 90°.
Volume	5575(9) $Å^3$
Z, Calculated density	8, 1.348 Mg/m^3
Absorption coefficient	0.087 mm^{-1}
F(000)	2368
Crystal size	0.75 x 0.35 x 0.06 mm
Theta range for data collection	1.92 to 25.05°.
Limiting indices	-13<=h<=8, -7<=k<=10, -66<=l<=65
Reflections collected / unique	14399 / 4801 [R(int) = 0.0444]
Completeness to theta = 25.00	97.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9948 and 0.9377
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4801 / 0 / 388
Goodness-of-fit on F^2	1.098
Final R indices [I>2sigma(I)]	R1 = 0.0643, wR2 = 0.1336
R indices (all data)	R1 = 0.0994, wR2 = 0.1463
Largest diff. peak and hole	0.282 and -0.255 $e.Å^{-3}$

Table A2.71. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for pzt, **1.71.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
N(1)	6956(2)	1730(3)	2083(1)	32(1)
N(1')	3973(2)	3023(3)	1974(1)	28(1)
N(1")	1431(2)	4395(4)	1653(1)	39(1)
N(01)	9843(2)	5799(3)	532(1)	32(1)
N(02)	8888(2)	5674(4)	385(1)	44(1)
N(01')	10934(2)	8042(3)	414(1)	34(1)
N(02')	10518(3)	9227(4)	283(1)	50(1)
N(01")	10947(2)	7227(3)	812(1)	31(1)
N(02")	11848(2)	8249(4)	850(1)	40(1)
O	8506(2)	7670(3)	833(1)	48(1)
C(2)	5812(3)	2031(4)	2129(1)	27(1)
C(3)	5243(3)	1433(4)	2327(1)	34(1)
C(4)	5866(3)	490(4)	2480(1)	40(1)
C(5)	7040(3)	153(4)	2432(1)	39(1)
C(6)	7538(3)	812(4)	2234(1)	36(1)
C(2')	5159(2)	3003(4)	1953(1)	26(1)
C(3')	5758(3)	3816(4)	1777(1)	27(1)
C(4')	5132(3)	4725(4)	1615(1)	25(1)
C(5')	3904(2)	4704(4)	1636(1)	27(1)
C(6')	3361(2)	3842(4)	1813(1)	25(1)
C(2")	2043(2)	3762(4)	1831(1)	28(1)
C(3")	1494(3)	3046(4)	2020(1)	34(1)
C(4")	274(3)	2994(4)	2028(1)	39(1)
C(5")	-364(3)	3625(4)	1846(1)	39(1)
C(6")	246(3)	4307(5)	1663(1)	43(1)
C(1"')	5733(2)	5656(4)	1430(1)	26(1)
C(2"')	5144(3)	6081(4)	1224(1)	29(1)
C(3"')	5693(3)	6985(4)	1054(1)	32(1)
C(4"')	6850(3)	7500(4)	1084(1)	29(1)
C(5"')	7446(3)	7094(4)	1290(1)	32(1)
C(6"')	6895(3)	6191(4)	1460(1)	29(1)
C(7)	7458(3)	8439(4)	895(1)	38(1)
C(01)	9134(3)	8409(4)	649(1)	36(1)
C(02)	10214(3)	7378(4)	600(1)	30(1)
C(03)	8933(3)	4204(5)	311(1)	52(1)
C(04)	9888(3)	3375(5)	403(1)	45(1)
C(05)	10474(3)	4434(4)	539(1)	36(1)
C(03')	11338(3)	9388(5)	115(1)	49(1)
C(04')	12248(3)	8356(5)	137(1)	45(1)
C(05')	11972(3)	7474(5)	330(1)	47(1)
C(03")	12161(3)	7966(4)	1073(1)	40(1)
C(04")	11466(3)	6830(4)	1178(1)	39(1)
C(05")	10676(3)	6375(4)	1010(1)	38(1)

Table A2.72. Selected bond lengths [Å] and angles [°] for pzt, **1.71.**

N(1)-C(6)	1.340(4)
N(1)-C(2)	1.358(4)
N(1')-C(6')	1.348(4)
N(1')-C(2')	1.361(4)
N(1")-C(2")	1.338(4)
N(1")-C(6")	1.358(4)
N(01)-N(02)	1.369(4)
N(01)-C(05)	1.392(4)
N(01)-C(02)	1.490(4)
N(02)-C(03)	1.349(5)
N(01')-N(02')	1.354(4)
N(01')-C(05')	1.370(4)
N(01')-C(02)	1.447(4)
N(02')-C(03')	1.333(4)
N(01")-C(05")	1.369(4)
N(01")-N(02")	1.379(4)
N(01")-C(02)	1.457(4)
N(02")-C(03")	1.320(4)
O-C(01)	1.414(4)
O-C(7)	1.415(4)

Symmetry transformations used to generate equivalent atoms:

Table A2.73. Bond lengths [Å] and angles [°] for pzt, **1.71.**

N(1)-C(6)	1.340(4)
N(1)-C(2)	1.358(4)
N(1')-C(6')	1.348(4)
N(1')-C(2')	1.361(4)
N(1")-C(2")	1.338(4)
N(1")-C(6")	1.358(4)
N(01)-N(02)	1.369(4)
N(01)-C(05)	1.392(4)
N(01)-C(02)	1.490(4)
N(02)-C(03)	1.349(5)
N(01')-N(02')	1.354(4)
N(01')-C(05')	1.370(4)
N(01')-C(02)	1.447(4)
N(02')-C(03')	1.333(4)
N(01")-C(05")	1.369(4)
N(01")-N(02")	1.379(4)
N(01")-C(02)	1.457(4)
N(02")-C(03")	1.320(4)
O-C(01)	1.414(4)
O-C(7)	1.415(4)
C(2)-C(3)	1.390(4)
C(2)-C(2')	1.499(4)
C(3)-C(4)	1.384(5)
C(3)-H(3)	0.9500
C(4)-C(5)	1.400(5)
C(4)-H(4)	0.9500
C(5)-C(6)	1.374(5)
C(5)-H(5)	0.9500
C(6)-H(6)	0.9500
C(2')-C(3')	1.390(4)
C(3')-C(4')	1.399(4)
C(3')-H(3')	0.9500
C(4')-C(5')	1.409(4)
C(4')-C(1"')	1.484(4)
C(5')-C(6')	1.389(4)
C(5')-H(4')	0.9500
C(6')-C(2")	1.511(4)
C(2")-C(3")	1.379(4)
C(3")-C(4")	1.395(4)
C(3")-H(3")	0.9500
C(4")-C(5")	1.367(5)
C(4")-H(4")	0.9500
C(5")-C(6")	1.372(5)
C(5")-H(5")	0.9500
C(6")-H(6")	0.9500
C(1"')-C(2"')	1.387(4)
C(1"')-C(6"')	1.417(4)
C(2"')-C(3"')	1.387(4)
C(2"')-H(2"')	0.9500
C(3"')-C(4"')	1.407(4)
C(3"')-H(3"')	0.9500
C(4"')-C(5"')	1.384(4)
C(4"')-C(7)	1.508(4)
C(5"')-C(6"')	1.386(4)
C(5"')-H(5"')	0.9500
C(6"')-H(6"')	0.9500
C(7)-H(7A)	0.9900

C(7)-H(7B)	0.9900
C(01)-C(02)	1.551(5)
C(01)-H(01A)	0.9900
C(01)-H(01B)	0.9900
C(03)-C(04)	1.407(5)
C(03)-H(03)	0.9500
C(04)-C(05)	1.369(5)
C(04)-H(04)	0.9500
C(05)-H(05)	0.9500
C(03')-C(04')	1.381(5)
C(03')-H(03')	0.9500
C(04')-C(05')	1.363(5)
C(04')-H(04')	0.9500
C(05')-H(05')	0.9500
C(03")-C(04")	1.399(5)
C(03")-H(03")	0.9500
C(04")-C(05")	1.364(5)
C(04")-H(04")	0.9500
C(05")-H(05")	0.9500

C(6)-N(1)-C(2)	118.3(3)
C(6')-N(1')-C(2')	117.7(3)
C(2")-N(1")-C(6")	117.9(3)
N(02)-N(01)-C(05)	111.2(3)
N(02)-N(01)-C(02)	117.0(3)
C(05)-N(01)-C(02)	129.4(3)
C(03)-N(02)-N(01)	103.3(3)
N(02')-N(01')-C(05')	113.1(3)
N(02')-N(01')-C(02)	119.7(3)
C(05')-N(01')-C(02)	126.6(3)
C(03')-N(02')-N(01')	102.4(3)
C(05")-N(01")-N(02")	113.0(3)
C(05")-N(01")-C(02)	125.2(3)
N(02")-N(01")-C(02)	120.0(3)
C(03")-N(02")-N(01")	103.3(3)
C(01)-O-C(7)	113.0(2)
N(1)-C(2)-C(3)	121.9(3)
N(1)-C(2)-C(2')	117.7(3)
C(3)-C(2)-C(2')	120.3(3)
C(4)-C(3)-C(2)	118.5(3)
C(4)-C(3)-H(3)	120.8
C(2)-C(3)-H(3)	120.8
C(3)-C(4)-C(5)	119.9(3)
C(3)-C(4)-H(4)	120.0
C(5)-C(4)-H(4)	120.0
C(6)-C(5)-C(4)	117.7(3)
C(6)-C(5)-H(5)	121.1
C(4)-C(5)-H(5)	121.1
N(1)-C(6)-C(5)	123.6(3)
N(1)-C(6)-H(6)	118.2
C(5)-C(6)-H(6)	118.2
N(1')-C(2')-C(3')	123.1(3)
N(1')-C(2')-C(2)	116.4(3)
C(3')-C(2')-C(2)	120.5(3)
C(2')-C(3')-C(4')	119.6(3)
C(2')-C(3')-H(3')	120.2
C(4')-C(3')-H(3')	120.2
C(3')-C(4')-C(5')	116.7(3)
C(3')-C(4')-C(1"')	121.6(3)
C(5')-C(4')-C(1"')	121.6(3)
C(6')-C(5')-C(4')	120.7(3)

C(6')-C(5')-H(4')	119.7
C(4')-C(5')-H(4')	119.7
N(1')-C(6')-C(5')	122.2(3)
N(1')-C(6')-C(2")	116.5(3)
C(5')-C(6')-C(2")	121.4(3)
N(1")-C(2")-C(3")	121.3(3)
N(1")-C(2")-C(6')	116.7(3)
C(3")-C(2")-C(6')	121.9(3)
C(2")-C(3")-C(4")	119.5(3)
C(2")-C(3")-H(3")	120.3
C(4")-C(3")-H(3")	120.3
C(5")-C(4")-C(3")	119.9(3)
C(5")-C(4")-H(4")	120.1
C(3")-C(4")-H(4")	120.1
C(4")-C(5")-C(6")	117.2(3)
C(4")-C(5")-H(5")	121.4
C(6")-C(5")-H(5")	121.4
N(1")-C(6")-C(5")	124.2(3)
N(1")-C(6")-H(6")	117.9
C(5")-C(6")-H(6")	117.9
C(2"')-C(1"')-C(6"')	117.6(3)
C(2"')-C(1"')-C(4')	120.2(3)
C(6"')-C(1"')-C(4')	122.2(3)
C(1"')-C(2"')-C(3"')	120.3(3)
C(1"')-C(2"')-H(2"')	119.8
C(3"')-C(2"')-H(2"')	119.8
C(2"')-C(3"')-C(4"')	121.6(3)
C(2"')-C(3"')-H(3"')	119.2
C(4"')-C(3"')-H(3"')	119.2
C(5"')-C(4"')-C(3"')	118.8(3)
C(5"')-C(4"')-C(7)	119.8(3)
C(3"')-C(4"')-C(7)	121.4(3)
C(4"')-C(5"')-C(6"')	119.5(3)
C(4"')-C(5"')-H(5"')	120.2
C(6"')-C(5"')-H(5"')	120.2
C(5"')-C(6"')-C(1"')	122.2(3)
C(5"')-C(6"')-H(6"')	118.9
C(1"')-C(6"')-H(6"')	118.9
O-C(7)-C(4"')	107.6(3)
O-C(7)-H(7A)	110.2
C(4"')-C(7)-H(7A)	110.2
O-C(7)-H(7B)	110.2
C(4"')-C(7)-H(7B)	110.2
H(7A)-C(7)-H(7B)	108.5
O-C(01)-C(02)	105.5(3)
O-C(01)-H(01A)	110.6
C(02)-C(01)-H(01A)	110.6
O-C(01)-H(01B)	110.6
C(02)-C(01)-H(01B)	110.6
H(01A)-C(01)-H(01B)	108.8
N(01')-C(02)-N(01")	107.0(2)
N(01')-C(02)-N(01)	110.3(2)
N(01")-C(02)-N(01)	106.8(2)
N(01')-C(02)-C(01)	110.3(3)
N(01")-C(02)-C(01)	111.5(2)
N(01)-C(02)-C(01)	110.7(3)
N(02)-C(03)-C(04)	113.8(3)
N(02)-C(03)-H(03)	123.1
C(04)-C(03)-H(03)	123.1
C(05)-C(04)-C(03)	103.8(3)
C(05)-C(04)-H(04)	128.1

C(03)-C(04)-H(04)	128.1
C(04)-C(05)-N(01)	107.8(3)
C(04)-C(05)-H(05)	126.1
N(01)-C(05)-H(05)	126.1
N(02')-C(03')-C(04')	113.7(3)
N(02')-C(03')-H(03')	123.2
C(04')-C(03')-H(03')	123.2
C(05')-C(04')-C(03')	105.1(3)
C(05')-C(04')-H(04')	127.4
C(03')-C(04')-H(04')	127.4
C(04')-C(05')-N(01')	105.7(3)
C(04')-C(05')-H(05')	127.2
N(01')-C(05')-H(05')	127.2
N(02")-C(03")-C(04")	112.0(3)
N(02")-C(03")-H(03")	124.0
C(04")-C(03")-H(03")	124.0
C(05")-C(04")-C(03")	106.9(3)
C(05")-C(04")-H(04")	126.6
C(03")-C(04")-H(04")	126.6
C(04")-C(05")-N(01")	104.6(3)
C(04")-C(05")-H(05")	127.7
N(01")-C(05")-H(05")	127.7

Symmetry transformations used to generate equivalent atoms:

Table A2.74. Anisotropic displacement parameters (A^2 x 10^3) for pzt, **1.71.**The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
N(1)	28(1)	37(2)	29(1)	1(1)	-3(1)	1(1)
N(1')	26(1)	32(2)	25(1)	0(1)	1(1)	-1(1)
N(1")	25(1)	60(2)	32(2)	5(1)	-3(1)	1(1)
N(01)	30(1)	37(2)	28(1)	0(1)	-2(1)	-1(1)
N(02)	40(2)	57(2)	36(2)	0(2)	-10(1)	-2(2)
N(01')	38(2)	41(2)	23(1)	7(1)	4(1)	1(1)
N(02')	46(2)	57(2)	45(2)	22(2)	6(1)	5(2)
N(01")	33(1)	36(2)	24(1)	1(1)	0(1)	-3(1)
N(02")	35(2)	45(2)	41(2)	2(1)	-1(1)	-7(2)
O	49(1)	52(2)	42(1)	21(1)	22(1)	19(1)
C(2)	31(2)	27(2)	22(2)	-2(1)	-1(1)	-2(2)
C(3)	35(2)	38(2)	28(2)	1(2)	2(1)	4(2)
C(4)	50(2)	41(2)	28(2)	5(2)	3(2)	1(2)
C(5)	46(2)	40(2)	32(2)	6(2)	-10(2)	4(2)
C(6)	30(2)	39(2)	37(2)	-1(2)	-6(2)	3(2)
C(2')	26(2)	30(2)	21(2)	-2(1)	1(1)	0(2)
C(3')	24(2)	32(2)	25(2)	-3(1)	0(1)	-1(1)
C(4')	26(2)	30(2)	19(1)	-3(1)	0(1)	-2(1)
C(5')	25(2)	34(2)	21(2)	-1(1)	-3(1)	1(2)
C(6')	24(2)	29(2)	22(2)	-4(1)	2(1)	0(1)
C(2")	24(2)	33(2)	27(2)	-5(1)	-1(1)	-1(2)
C(3")	27(2)	45(2)	30(2)	0(2)	3(1)	-3(2)
C(4")	33(2)	46(2)	36(2)	-5(2)	8(1)	-5(2)
C(5")	22(2)	50(2)	44(2)	-14(2)	2(2)	-1(2)
C(6")	28(2)	65(3)	35(2)	2(2)	-6(1)	3(2)
C(1"')	27(2)	27(2)	25(2)	0(1)	1(1)	3(2)
C(2"')	23(2)	37(2)	28(2)	1(2)	0(1)	1(2)
C(3"')	34(2)	37(2)	25(2)	6(2)	2(1)	3(2)
C(4"')	31(2)	25(2)	30(2)	3(1)	7(1)	1(2)
C(5"')	27(2)	33(2)	36(2)	2(2)	3(1)	-6(2)
C(6"')	30(2)	35(2)	22(2)	1(1)	-1(1)	-1(2)
C(7)	34(2)	36(2)	44(2)	12(2)	8(2)	2(2)
C(01)	46(2)	36(2)	25(2)	10(2)	9(1)	3(2)
C(02)	31(2)	36(2)	23(2)	4(1)	3(1)	-1(2)
C(03)	60(3)	55(3)	40(2)	-8(2)	-10(2)	-11(2)
C(04)	50(2)	40(2)	45(2)	-7(2)	4(2)	-3(2)
C(05)	31(2)	42(2)	36(2)	2(2)	2(1)	2(2)
C(03')	45(2)	65(3)	38(2)	22(2)	4(2)	-12(2)
C(04')	48(2)	56(3)	32(2)	-8(2)	11(2)	-14(2)
C(05')	49(2)	49(2)	44(2)	6(2)	16(2)	5(2)
C(03")	39(2)	42(2)	37(2)	-4(2)	-11(2)	1(2)
C(04")	45(2)	44(2)	28(2)	4(2)	-4(2)	7(2)
C(05")	41(2)	44(2)	28(2)	7(2)	2(1)	-2(2)

Table A2.75. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for pzt, **1.71.**

	x	y	z	U(eq)
H(3)	4444	1666	2357	40
H(4)	5497	72	2618	48
H(5)	7478	-508	2533	47
H(6)	8339	605	2201	43
H(3')	6586	3755	1767	32
H(4')	3442	5285	1527	32
H(3")	1942	2593	2145	41
H(4")	-112	2520	2159	46
H(5")	-1195	3591	1846	46
H(6")	-189	4747	1536	51
H(2"')	4361	5752	1199	35
H(3"')	5277	7262	913	38
H(5"')	8228	7431	1314	38
H(6"')	7310	5923	1601	35
H(7A)	6950	8543	752	45
H(7B)	7635	9478	956	45
H(01A)	9382	9448	699	43
H(01B)	8646	8503	503	43
H(03)	8373	3768	205	62
H(04)	10080	2327	378	54
H(05)	11186	4267	622	44
H(03')	11300	10142	-7	59
H(04')	12922	8277	38	54
H(05')	12409	6639	392	57
H(03")	12785	8474	1153	47
H(04")	11531	6449	1337	47
H(05")	10073	5633	1026	45

Table A2.76. Selected torsion angles [°] for pzt, **1.71**.

C(3')–C(4')–C(1"')–C(2"')	156.4(3)
C(5')–C(4')–C(1"')–C(2"')	–23.2(5)
C(3')–C(4')–C(1"')–C(6"')	–26.1(5)
C(5')–C(4')–C(1"')–C(6"')	154.3(3)
C(01)–O–C(7)–C(4"')	177.4(3)
C(5"')–C(4"')–C(7)–O	53.9(4)
C(3"')–C(4"')–C(7)–O	–124.2(3)
C(7)–O–C(01)–C(02)	–177.8(3)

Symmetry transformations used to generate equivalent atoms:

Table A2.77. Torsion angles [°] for pzt, **1.71.**

C(05)–N(01)–N(02)–C(03)	1.9(4)
C(02)–N(01)–N(02)–C(03)	166.0(3)
C(05')–N(01')–N(02')–C(03')	-0.7(4)
C(02)–N(01')–N(02')–C(03')	-172.4(3)
C(05")–N(01")–N(02")–C(03")	2.5(4)
C(02)–N(01")–N(02")–C(03")	168.2(3)
C(6)–N(1)–C(2)–C(3)	0.9(5)
C(6)–N(1)–C(2)–C(2')	-177.4(3)
N(1)–C(2)–C(3)–C(4)	-0.8(5)
C(2')–C(2)–C(3)–C(4)	177.4(3)
C(2)–C(3)–C(4)–C(5)	-0.3(5)
C(3)–C(4)–C(5)–C(6)	1.3(5)
C(2)–N(1)–C(6)–C(5)	0.2(5)
C(4)–C(5)–C(6)–N(1)	-1.2(5)
C(6')–N(1')–C(2')–C(3')	1.7(5)
C(6')–N(1')–C(2')–C(2)	-177.6(3)
N(1)–C(2)–C(2')–N(1')	166.6(3)
C(3)–C(2)–C(2')–N(1')	-11.7(4)
N(1)–C(2)–C(2')–C(3')	-12.7(4)
C(3)–C(2)–C(2')–C(3')	169.0(3)
N(1')–C(2')–C(3')–C(4')	1.1(5)
C(2)–C(2')–C(3')–C(4')	-179.6(3)
C(2')–C(3')–C(4')–C(5')	-2.6(4)
C(2')–C(3')–C(4')–C(1"')	177.8(3)
C(3')–C(4')–C(5')–C(6')	1.4(4)
C(1"')–C(4')–C(5')–C(6')	-179.0(3)
C(2')–N(1')–C(6')–C(5')	-3.0(4)
C(2')–N(1')–C(6')–C(2")	176.3(3)
C(4')–C(5')–C(6')–N(1')	1.4(5)
C(4')–C(5')–C(6')–C(2")	-177.8(3)
C(6")–N(1")–C(2")–C(3")	-0.2(5)
C(6")–N(1")–C(2")–C(6')	178.5(3)
N(1')–C(6')–C(2")–N(1")	-171.0(3)
C(5')–C(6')–C(2")–N(1")	8.2(5)
N(1')–C(6')–C(2")–C(3")	7.7(5)
C(5')–C(6')–C(2")–C(3")	-173.0(3)
N(1")–C(2")–C(3")–C(4")	-0.7(5)
C(6')–C(2")–C(3")–C(4")	-179.4(3)
C(2")–C(3")–C(4")–C(5")	1.2(5)
C(3")–C(4")–C(5")–C(6")	-0.8(5)
C(2")–N(1")–C(6")–C(5")	0.7(6)
C(4")–C(5")–C(6")–N(1")	-0.2(6)
C(3')–C(4')–C(1"')–C(2"')	156.4(3)
C(5')–C(4')–C(1"')–C(2"')	-23.2(5)
C(3')–C(4')–C(1"')–C(6"')	-26.1(5)
C(5')–C(4')–C(1"')–C(6"')	154.3(3)
C(6"')–C(1"')–C(2"')–C(3"')	0.7(5)
C(4')–C(1"')–C(2"')–C(3"')	178.3(3)
C(1"')–C(2"')–C(3"')–C(4"')	-0.2(5)
C(2"')–C(3"')–C(4"')–C(5"')	-0.2(5)
C(2"')–C(3"')–C(4"')–C(7)	178.0(3)
C(3"')–C(4"')–C(5"')–C(6"')	0.2(5)
C(7)–C(4"')–C(5"')–C(6"')	-178.0(3)
C(4"')–C(5"')–C(6"')–C(1"')	0.3(5)
C(2"')–C(1"')–C(6"')–C(5"')	-0.8(5)
C(4')–C(1"')–C(6"')–C(5"')	-178.3(3)
C(01)–O–C(7)–C(4"')	177.4(3)

C(5"')-C(4"')-C(7)-O	53.9(4)
C(3"')-C(4"')-C(7)-O	-124.2(3)
C(7)-O-C(01)-C(02)	-177.8(3)
N(02')-N(01')-C(02)-N(01")	-133.1(3)
C(05')-N(01')-C(02)-N(01")	56.4(4)
N(02')-N(01')-C(02)-N(01)	111.1(3)
C(05')-N(01')-C(02)-N(01)	-59.4(4)
N(02')-N(01')-C(02)-C(01)	-11.6(4)
C(05')-N(01')-C(02)-C(01)	177.9(3)
C(05")-N(01")-C(02)-N(01')	-166.1(3)
N(02")-N(01")-C(02)-N(01')	30.1(4)
C(05")-N(01")-C(02)-N(01)	-47.9(4)
N(02")-N(01")-C(02)-N(01)	148.3(3)
C(05")-N(01")-C(02)-C(01)	73.2(4)
N(02")-N(01")-C(02)-C(01)	-90.6(3)
N(02)-N(01)-C(02)-N(01')	-82.9(3)
C(05)-N(01)-C(02)-N(01')	77.8(4)
N(02)-N(01)-C(02)-N(01")	161.1(2)
C(05)-N(01)-C(02)-N(01")	-38.2(4)
N(02)-N(01)-C(02)-C(01)	39.5(3)
C(05)-N(01)-C(02)-C(01)	-159.8(3)
O-C(01)-C(02)-N(01')	-179.2(3)
O-C(01)-C(02)-N(01")	-60.4(3)
O-C(01)-C(02)-N(01)	58.3(3)
N(01)-N(02)-C(03)-C(04)	-0.2(4)
N(02)-C(03)-C(04)-C(05)	-1.6(4)
C(03)-C(04)-C(05)-N(01)	2.6(4)
N(02)-N(01)-C(05)-C(04)	-3.0(4)
C(02)-N(01)-C(05)-C(04)	-164.6(3)
N(01')-N(02')-C(03')-C(04')	0.0(4)
N(02')-C(03')-C(04')-C(05')	0.7(5)
C(03')-C(04')-C(05')-N(01')	-1.0(4)
N(02')-N(01')-C(05')-C(04')	1.1(4)
C(02)-N(01')-C(05')-C(04')	172.1(3)
N(01")-N(02")-C(03")-C(04")	-1.7(4)
N(02")-C(03")-C(04")-C(05")	0.3(4)
C(03")-C(04")-C(05")-N(01")	1.2(4)
N(02")-N(01")-C(05")-C(04")	-2.4(4)
C(02)-N(01")-C(05")-C(04")	-167.2(3)

Symmetry transformations used to generate equivalent atoms:

Table A2.78. Hydrogen bonds for pzt, **1.71** [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

Table A2.79. Crystal data and structure refinement for $[Ag_2(pzt)_2](BF_4)_2$, **4.2.**

Identification code	**4.2**
Empirical formula	C66 H54 Ag2 B2 F8 N18 O2
Formula weight	1520.63
Temperature	569(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, *p* 21/*c*
Unit cell dimensions	a = 8.5329(16) Å alpha = 90°. b = 22.108(5) Å beta = 96.534(3)°. c = 17.393(4) Å gamma = 90°.
Volume	3259.9(11) $Å^3$
Z, Calculated density	2, 1.549 Mg/m^3
Absorption coefficient	0.684 mm^{-1}
F(000)	1536
Crystal size	0.45 x 0.08 x 0.07 mm
Theta range for data collection	2.19 to 26.41°.
Limiting indices	-10<=h<=10, -27<=k<=25, -21<=l<=21
Reflections collected / unique	28177 / 6637 [R(int) = 0.1203]
Completeness to theta = 26.41	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9537 and 0.7482
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6637 / 20 / 480
Goodness-of-fit on F^2	0.955
Final R indices [I>2sigma(I)]	R1 = 0.0503, wR2 = 0.0835
R indices (all data)	R1 = 0.1633, wR2 = 0.1095
Largest diff. peak and hole	0.311 and -0.367 $eÅ^{-3}$

Table A2.80. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $[Ag_2(pzt)_2](BF_4)_2$, **4.2.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ag	6291(1)	-635(1)	12329(1)	67(1)
N(1)	4809(6)	255(2)	12883(3)	92(2)
N(1')	4332(4)	-214(2)	11448(2)	45(1)
N(1")	5636(4)	-1315(2)	11287(2)	51(1)
N(01)	719(4)	771(2)	6294(2)	48(1)
N(02)	1612(4)	432(2)	6819(2)	53(1)
N(01')	49(4)	1669(2)	5594(2)	49(1)
N(02')	558(5)	1491(2)	4915(2)	76(1)
N(01")	2565(4)	1566(2)	6333(2)	53(1)
N(02")	3211(5)	2019(2)	6787(2)	70(1)
O	-1259(3)	1441(1)	7067(2)	50(1)
C(2)	3957(5)	577(2)	12366(3)	59(1)
C(3)	3361(9)	1124(3)	12542(4)	135(3)
C(4)	3683(11)	1349(4)	13280(4)	157(4)
C(5)	4623(8)	1036(4)	13805(4)	106(2)
C(6)	5111(7)	493(4)	13594(4)	117(3)
C(2')	3610(5)	311(2)	11573(3)	48(1)
C(3')	2594(5)	586(2)	11007(2)	50(1)
C(4')	2270(5)	328(2)	10277(2)	42(1)
C(5')	2995(5)	-222(2)	10160(2)	45(1)
C(6')	3991(5)	-485(2)	10754(2)	41(1)
C(2")	4747(5)	-1088(2)	10663(3)	44(1)
C(3")	4548(6)	-1404(2)	9979(3)	64(1)
C(4")	5279(7)	-1954(3)	9918(3)	78(2)
C(5")	6170(6)	-2186(2)	10549(3)	70(2)
C(6")	6306(6)	-1858(2)	11213(3)	64(1)
C(1"')	1201(5)	622(2)	9646(2)	44(1)
C(6"')	667(6)	1206(2)	9731(3)	64(1)
C(5"')	-287(6)	1488(2)	9141(3)	70(2)
C(4"')	-760(5)	1203(2)	8458(3)	49(1)
C(3"')	-262(5)	618(2)	8375(2)	54(1)
C(2"')	708(5)	333(2)	8955(3)	48(1)
C(7)	-1791(5)	1524(2)	7815(2)	63(1)
C(01)	249(5)	1692(2)	7022(2)	45(1)
C(02)	899(5)	1436(2)	6306(2)	43(1)
C(03)	976(7)	-121(3)	6739(3)	68(2)
C(04)	-307(7)	-132(3)	6186(3)	76(2)
C(05)	-453(6)	447(3)	5912(3)	70(2)
C(03')	-541(7)	1715(2)	4387(3)	74(2)
C(04')	-1692(6)	2027(2)	4709(3)	67(1)
C(05')	-1287(5)	1992(2)	5486(3)	53(1)
C(03")	4742(8)	1993(3)	6683(4)	89(2)
C(04")	5048(7)	1539(4)	6195(4)	98(2)
C(05")	3650(7)	1271(3)	5976(3)	86(2)
B	8518(4)	1577(1)	1881(2)	79(2)
F(1A)	7567(13)	2046(4)	1868(7)	136(7)
F(2A)	9418(12)	1627(6)	1318(5)	174(8)
F(3A)	9410(12)	1558(6)	2549(4)	151(6)
F(4A)	7680(15)	1078(4)	1788(7)	187(9)
F(1B)	8460(20)	1583(5)	2634(2)	288(11)
F(2B)	7551(12)	1990(3)	1553(7)	111(5)
F(3B)	8093(14)	1040(3)	1604(6)	162(8)
F(4B)	9964(8)	1696(5)	1732(11)	273(13)

Table A2.81. Selected bond lengths [Å] and angles [°] for $[Ag_2(pzt)_2](BF_4)_2$, **4.2.**

Ag–N(02)#1	2.236(4)
Ag–N(1')	2.329(3)
Ag–N(1")	2.372(4)
Ag–N(1)	2.586(4)
N(02)–Ag#1	2.236(4)
N(02)#1–Ag–N(1')	144.67(14)
N(02)#1–Ag–N(1")	137.33(13)
N(1')–Ag–N(1")	70.27(13)
N(02)#1–Ag–N(1)	89.31(14)
N(1')–Ag–N(1)	66.16(14)
N(1")–Ag–N(1)	133.25(14)
C(2)–N(1)–Ag	114.6(3)
C(6)–N(1)–Ag	126.3(4)
C(6')–N(1')–Ag	118.4(3)
C(6")–N(1")–Ag	125.0(3)
C(2")–N(1")–Ag	116.1(3)
C(03)–N(02)–Ag#1	122.5(3)
N(01)–N(02)–Ag#1	133.3(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y,-z+2

Table A2.82. Bond lengths [Å] and angles [°] for $[Ag_2(pzt)_2](BF_4)_2$, **4.2.**

Ag–N(02)#1	2.236(4)
Ag–N(1')	2.329(3)
Ag–N(1")	2.372(4)
Ag–N(1)	2.586(4)
N(1)–C(2)	1.302(6)
N(1)–C(6)	1.340(7)
N(1')–C(2')	1.342(5)
N(1')–C(6')	1.350(5)
N(1")–C(6")	1.342(5)
N(1")–C(2")	1.348(5)
N(01)–C(05)	1.343(5)
N(01)–N(02)	1.349(4)
N(01)–C(02)	1.478(5)
N(02)–C(03)	1.339(6)
N(02)–Ag#1	2.236(4)
N(01')–C(05')	1.340(5)
N(01')–N(02')	1.361(5)
N(01')–C(02)	1.456(5)
N(02')–C(03')	1.331(6)
N(01")–C(05")	1.342(6)
N(01")–N(02")	1.354(5)
N(01")–C(02)	1.446(5)
N(02")–C(03")	1.340(7)
O–C(01)	1.411(4)
O–C(7)	1.438(5)
C(2)–C(3)	1.361(7)
C(2)–C(2')	1.497(6)
C(3)–C(4)	1.376(8)
C(3)–H(3)	0.9300
C(4)–C(5)	1.338(8)
C(4)–H(4)	0.9300
C(5)–C(6)	1.336(8)
C(5)–H(5)	0.9300
C(6)–H(6)	0.9300
C(2')–C(3')	1.378(5)
C(3')–C(4')	1.391(5)
C(3')–H(3')	0.9300
C(4')–C(5')	1.390(6)
C(4')–C(1"')	1.493(5)
C(5')–C(6')	1.389(5)
C(5')–H(5')	0.9300
C(6')–C(2")	1.497(6)
C(2")–C(3")	1.373(6)
C(3")–C(4")	1.377(6)
C(3")–H(3")	0.9300
C(4")–C(5")	1.362(6)
C(4")–H(4")	0.9300
C(5")–C(6")	1.358(6)
C(5")–H(5")	0.9300
C(6")–H(6")	0.9300
C(1"')–C(6"')	1.382(6)
C(1"')–C(2"')	1.383(6)
C(6"')–C(5"')	1.383(6)
C(6"')–H(6"')	0.9300
C(5"')–C(4"')	1.365(6)
C(5"')–H(5"')	0.9300
C(4"')–C(3"')	1.374(6)

C(4"')-C(7)	1.518(6)
C(3"')-C(2"')	1.382(5)
C(3"')-H(3"')	0.9300
C(2"')-H(2"')	0.9300
C(7)-H(7A)	0.9700
C(7)-H(7B)	0.9700
C(01)-C(02)	1.529(5)
C(01)-H(01A)	0.9700
C(01)-H(01B)	0.9700
C(03)-C(04)	1.373(6)
C(03)-H(03)	0.9300
C(04)-C(05)	1.365(6)
C(04)-H(04)	0.9300
C(05)-H(05)	0.9300
C(03')-C(04')	1.371(7)
C(03')-H(03')	0.9300
C(04')-C(05')	1.359(6)
C(04')-H(04')	0.9300
C(05')-H(05')	0.9300
C(03")-C(04")	1.358(8)
C(03")-H(03")	0.9300
C(04")-C(05")	1.347(7)
C(04")-H(04")	0.9300
C(05")-H(05")	0.9300
B-F(4A)	1.315(2)
B-F(4B)	1.315(2)
B-F(1B)	1.316(2)
B-F(3A)	1.316(2)
B-F(1A)	1.316(2)
B-F(2B)	1.316(2)
B-F(3B)	1.316(2)
B-F(2A)	1.316(2)

N(02)#1-Ag-N(1')	144.67(14)
N(02)#1-Ag-N(1")	137.33(13)
N(1')-Ag-N(1")	70.27(13)
N(02)#1-Ag-N(1)	89.31(14)
N(1')-Ag-N(1)	66.16(14)
N(1")-Ag-N(1)	133.25(14)
C(2)-N(1)-C(6)	117.2(5)
C(2)-N(1)-Ag	114.6(3)
C(6)-N(1)-Ag	126.3(4)
C(2')-N(1')-C(6')	118.2(3)
C(2')-N(1')-Ag	123.2(3)
C(6')-N(1')-Ag	118.4(3)
C(6")-N(1")-C(2")	117.6(4)
C(6")-N(1")-Ag	125.0(3)
C(2")-N(1")-Ag	116.1(3)
C(05)-N(01)-N(02)	111.7(4)
C(05)-N(01)-C(02)	127.5(4)
N(02)-N(01)-C(02)	119.6(3)
C(03)-N(02)-N(01)	104.2(4)
C(03)-N(02)-Ag#1	122.5(3)
N(01)-N(02)-Ag#1	133.3(3)
C(05')-N(01')-N(02')	112.5(4)
C(05')-N(01')-C(02)	129.8(4)
N(02')-N(01')-C(02)	117.3(4)
C(03')-N(02')-N(01')	102.8(4)
C(05")-N(01")-N(02")	111.9(4)
C(05")-N(01")-C(02)	128.0(5)
N(02")-N(01")-C(02)	120.0(4)

C(03")-N(02")-N(01")	103.2(5)
C(01)-O-C(7)	112.5(3)
N(1)-C(2)-C(3)	121.5(5)
N(1)-C(2)-C(2')	117.3(5)
C(3)-C(2)-C(2')	121.1(5)
C(2)-C(3)-C(4)	119.5(6)
C(2)-C(3)-H(3)	120.3
C(4)-C(3)-H(3)	120.3
C(5)-C(4)-C(3)	119.3(7)
C(5)-C(4)-H(4)	120.3
C(3)-C(4)-H(4)	120.3
C(4)-C(5)-C(6)	117.3(6)
C(4)-C(5)-H(5)	121.3
C(6)-C(5)-H(5)	121.3
C(5)-C(6)-N(1)	124.9(6)
C(5)-C(6)-H(6)	117.5
N(1)-C(6)-H(6)	117.5
N(1')-C(2')-C(3')	122.0(4)
N(1')-C(2')-C(2)	116.2(4)
C(3')-C(2')-C(2)	121.8(4)
C(2')-C(3')-C(4')	120.9(4)
C(2')-C(3')-H(3')	119.5
C(4')-C(3')-H(3')	119.5
C(5')-C(4')-C(3')	116.6(4)
C(5')-C(4')-C(1"')	121.2(4)
C(3')-C(4')-C(1"')	122.2(4)
C(4')-C(5')-C(6')	120.2(4)
C(4')-C(5')-H(5')	119.9
C(6')-C(5')-H(5')	119.9
N(1')-C(6')-C(5')	122.1(4)
N(1')-C(6')-C(2")	116.0(4)
C(5')-C(6')-C(2")	121.9(4)
N(1")-C(2")-C(3")	120.6(4)
N(1")-C(2")-C(6')	117.2(4)
C(3")-C(2")-C(6')	122.1(4)
C(2")-C(3")-C(4")	120.3(4)
C(2")-C(3")-H(3")	119.9
C(4")-C(3")-H(3")	119.9
C(5")-C(4")-C(3")	119.1(5)
C(5")-C(4")-H(4")	120.4
C(3")-C(4")-H(4")	120.4
C(6")-C(5")-C(4")	118.1(5)
C(6")-C(5")-H(5")	121.0
C(4")-C(5")-H(5")	121.0
N(1")-C(6")-C(5")	124.2(4)
N(1")-C(6")-H(6")	117.9
C(5")-C(6")-H(6")	117.9
C(6"')-C(1"')-C(2"')	116.9(4)
C(6"')-C(1"')-C(4')	120.7(4)
C(2"')-C(1"')-C(4')	122.5(4)
C(1"')-C(6"')-C(5"')	121.1(4)
C(1"')-C(6"')-H(6"')	119.5
C(5"')-C(6"')-H(6"')	119.5
C(4"')-C(5"')-C(6"')	121.8(5)
C(4"')-C(5"')-H(5"')	119.1
C(6"')-C(5"')-H(5"')	119.1
C(5"')-C(4"')-C(3"')	117.5(4)
C(5"')-C(4"')-C(7)	121.0(5)
C(3"')-C(4"')-C(7)	121.5(4)
C(4"')-C(3"')-C(2"')	121.3(4)
C(4"')-C(3"')-H(3"')	119.4

C(2"')-C(3"')-H(3"')	119.4
C(3"')-C(2"')-C(1"')	121.5(4)
C(3"')-C(2"')-H(2"')	119.3
C(1"')-C(2"')-H(2"')	119.3
O-C(7)-C(4"')	113.0(4)
O-C(7)-H(7A)	109.0
C(4"')-C(7)-H(7A)	109.0
O-C(7)-H(7B)	109.0
C(4"')-C(7)-H(7B)	109.0
H(7A)-C(7)-H(7B)	107.8
O-C(01)-C(02)	108.5(3)
O-C(01)-H(01A)	110.0
C(02)-C(01)-H(01A)	110.0
O-C(01)-H(01B)	110.0
C(02)-C(01)-H(01B)	110.0
H(01A)-C(01)-H(01B)	108.4
N(01")-C(02)-N(01')	110.5(3)
N(01")-C(02)-N(01)	107.4(3)
N(01')-C(02)-N(01)	107.4(3)
N(01")-C(02)-C(01)	110.3(4)
N(01')-C(02)-C(01)	111.7(3)
N(01)-C(02)-C(01)	109.4(3)
N(02)-C(03)-C(04)	111.7(5)
N(02)-C(03)-H(03)	124.1
C(04)-C(03)-H(03)	124.1
C(05)-C(04)-C(03)	105.2(5)
C(05)-C(04)-H(04)	127.4
C(03)-C(04)-H(04)	127.4
N(01)-C(05)-C(04)	107.1(5)
N(01)-C(05)-H(05)	126.4
C(04)-C(05)-H(05)	126.4
N(02')-C(03')-C(04')	112.8(5)
N(02')-C(03')-H(03')	123.6
C(04')-C(03')-H(03')	123.6
C(05')-C(04')-C(03')	105.3(4)
C(05')-C(04')-H(04')	127.3
C(03')-C(04')-H(04')	127.3
N(01')-C(05')-C(04')	106.6(4)
N(01')-C(05')-H(05')	126.7
C(04')-C(05')-H(05')	126.7
N(02")-C(03")-C(04")	112.1(6)
N(02")-C(03")-H(03")	123.9
C(04")-C(03")-H(03")	123.9
C(05")-C(04")-C(03")	105.9(6)
C(05")-C(04")-H(04")	127.0
C(03")-C(04")-H(04")	127.0
N(01")-C(05")-C(04")	106.9(6)
N(01")-C(05")-H(05")	126.5
C(04")-C(05")-H(05")	126.5
F(4A)-B-F(4B)	130.7(9)
F(4A)-B-F(1B)	92.9(8)
F(4B)-B-F(1B)	109.49(10)
F(4A)-B-F(3A)	109.50(10)
F(4B)-B-F(3A)	73.6(7)
F(1B)-B-F(3A)	37.2(7)
F(4A)-B-F(1A)	109.51(10)
F(4B)-B-F(1A)	115.5(10)
F(1B)-B-F(1A)	85.3(8)
F(3A)-B-F(1A)	109.47(10)
F(4A)-B-F(2B)	102.9(10)
F(4B)-B-F(2B)	109.45(10)

F(1B)-B-F(2B)	109.45(10)
F(3A)-B-F(2B)	132.5(8)
F(1A)-B-F(2B)	24.6(8)
F(4A)-B-F(3B)	22.3(8)
F(4B)-B-F(3B)	109.49(10)
F(1B)-B-F(3B)	109.46(10)
F(3A)-B-F(3B)	113.7(9)
F(1A)-B-F(3B)	124.1(9)
F(2B)-B-F(3B)	109.49(10)
F(4A)-B-F(2A)	109.50(10)
F(4B)-B-F(2A)	36.6(7)
F(1B)-B-F(2A)	146.1(7)
F(3A)-B-F(2A)	109.42(10)
F(1A)-B-F(2A)	109.43(10)
F(2B)-B-F(2A)	90.6(8)
F(3B)-B-F(2A)	87.9(8)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y,-z+2

Table A2.83. Anisotropic displacement parameters (A^2 x 10^3) for $[Ag_2(pzt)_2](BF_4)_2$, **4.2.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Ag	76(1)	74(1)	47(1)	3(1)	-15(1)	12(1)
N(1)	113(4)	115(4)	44(3)	-20(3)	-17(3)	55(3)
N(1')	47(2)	46(3)	39(2)	0(2)	-1(2)	0(2)
N(1")	55(2)	47(3)	50(3)	0(2)	-2(2)	7(2)
N(01)	53(2)	45(3)	44(2)	2(2)	-4(2)	5(2)
N(02)	63(3)	44(3)	51(3)	7(2)	-3(2)	12(2)
N(01')	63(3)	47(3)	36(2)	1(2)	-2(2)	6(2)
N(02')	106(4)	79(3)	40(3)	4(2)	0(2)	26(3)
N(01")	43(2)	66(3)	49(2)	9(2)	3(2)	-5(2)
N(02")	67(3)	75(3)	63(3)	17(2)	-18(2)	-22(2)
O	45(2)	65(2)	38(2)	10(2)	3(1)	0(2)
C(2)	63(3)	61(4)	52(3)	-16(3)	0(2)	7(3)
C(3)	216(8)	96(6)	80(5)	-41(4)	-50(5)	63(5)
C(4)	236(9)	130(7)	87(6)	-64(5)	-58(6)	70(6)
C(5)	112(5)	133(7)	70(5)	-49(5)	-5(4)	26(5)
C(6)	127(6)	160(7)	56(4)	-25(4)	-24(4)	68(5)
C(2')	51(3)	49(3)	41(3)	2(2)	1(2)	4(2)
C(3')	57(3)	46(3)	47(3)	-2(3)	5(2)	4(2)
C(4')	45(3)	39(3)	42(3)	6(2)	3(2)	2(2)
C(5')	52(3)	54(3)	29(3)	4(2)	1(2)	-3(2)
C(6')	41(2)	45(3)	37(3)	3(2)	6(2)	-1(2)
C(2")	45(3)	43(3)	41(3)	-1(2)	-1(2)	-5(2)
C(3")	78(4)	55(4)	54(3)	-5(3)	-15(3)	12(3)
C(4")	110(5)	58(4)	61(4)	-17(3)	-9(3)	12(3)
C(5")	91(4)	45(3)	68(4)	-6(3)	-12(3)	13(3)
C(6")	76(4)	48(4)	64(4)	2(3)	-14(3)	7(3)
C(1"')	47(2)	47(3)	38(3)	9(3)	6(2)	4(2)
C(6"')	85(4)	59(4)	47(3)	-4(3)	-2(3)	17(3)
C(5"')	91(4)	59(4)	59(4)	8(3)	4(3)	29(3)
C(4"')	52(3)	57(4)	40(3)	14(3)	10(2)	15(2)
C(3"')	59(3)	65(4)	38(3)	7(3)	3(2)	1(3)
C(2"')	60(3)	44(3)	41(3)	8(2)	4(2)	5(2)
C(7)	56(3)	88(4)	45(3)	18(3)	12(2)	17(3)
C(01)	49(3)	46(3)	39(3)	1(2)	3(2)	0(2)
C(02)	45(3)	48(3)	35(3)	7(2)	0(2)	0(2)
C(03)	97(4)	46(4)	60(4)	4(3)	5(3)	9(3)
C(04)	97(4)	42(4)	86(4)	-6(3)	-2(3)	-14(3)
C(05)	61(3)	71(4)	73(4)	-10(3)	-14(3)	-2(3)
C(03')	106(5)	75(4)	36(3)	9(3)	-9(3)	0(3)
C(04')	69(4)	64(4)	62(4)	24(3)	-11(3)	3(3)
C(05')	53(3)	50(3)	56(3)	13(3)	0(2)	0(2)
C(03")	59(4)	112(6)	89(5)	53(4)	-20(3)	-35(4)
C(04")	56(4)	124(6)	117(6)	47(5)	23(4)	-5(4)
C(05")	59(4)	104(5)	98(5)	6(4)	27(3)	-3(4)
B	90(6)	82(7)	58(6)	9(5)	-15(5)	16(5)
F(1A)	133(11)	86(8)	201(16)	-15(9)	75(9)	46(7)
F(2A)	239(15)	220(17)	80(7)	57(7)	92(9)	102(12)
F(3A)	135(10)	257(15)	53(8)	-4(7)	-26(5)	-14(7)
F(4A)	168(12)	153(17)	219(19)	75(13)	-74(10)	-51(10)
F(1B)	630(30)	169(13)	42(7)	-7(7)	-46(11)	-3(16)
F(2B)	150(10)	75(7)	100(7)	31(5)	-30(6)	29(6)
F(3B)	337(19)	37(8)	92(9)	-42(6)	-67(10)	58(9)
F(4B)	117(9)	226(16)	450(30)	-120(19)	-69(15)	69(9)

Table A2.84. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for $[Ag_2(pzt)_2](BF_4)_2$, **4.2.**

	x	y	z	U(eq)
H(3)	2741	1344	12166	163
H(4)	3251	1716	13413	188
H(5)	4924	1190	14297	127
H(6)	5705	263	13968	140
H(3')	2118	950	11114	60
H(5')	2812	-415	9683	54
H(3")	3918	-1245	9556	77
H(4")	5165	-2165	9452	94
H(5")	6671	-2558	10525	84
H(6")	6901	-2021	11645	77
H(6"')	954	1412	10191	77
H(5"')	-614	1883	9212	84
H(3"')	-583	410	7920	65
H(2"')	1036	-61	8880	58
H(7A)	-2862	1373	7800	75
H(7B)	-1807	1953	7931	75
H(01A)	948	1590	7483	54
H(01B)	174	2129	6985	54
H(03)	1357	-457	7023	82
H(04)	-940	-462	6031	91
H(05)	-1221	589	5533	84
H(03')	-530	1665	3857	88
H(04')	-2564	2222	4450	80
H(05')	-1830	2160	5868	64
H(03")	5505	2254	6917	107
H(04")	6023	1435	6043	117
H(05")	3472	944	5641	103

Table A2.85. Selected torsion angles [°] for $[Ag_2(pzt)_2](BF_4)_2$, **4.2**.

C(5')–C(4')–C(1"')–C(2"')	8.7(6)
C(3')–C(4')–C(1"')–C(2"')	–171.2(4)
C(01)–O–C(7)–C(4"')	–63.4(5)
C(5"')–C(4"')–C(7)–O	134.4(4)
C(3"')–C(4"')–C(7)–O	–45.2(6)
C(7)–O–C(01)–C(02)	164.5(3)

Symmetry transformations used to generate equivalent atoms:
#1 –x+1,–y,–z+2

Table A2.86. Torsion angles [°] for $[Ag_2(pzt)_2](BF_4)_2$, **4.2.**

N(02)#1-Ag-N(1)-C(2)	139.9(4)
N(1')-Ag-N(1)-C(2)	-13.9(4)
N(1")-Ag-N(1)-C(2)	-36.6(5)
N(02)#1-Ag-N(1)-C(6)	-23.6(6)
N(1')-Ag-N(1)-C(6)	-177.5(6)
N(1")-Ag-N(1)-C(6)	159.8(5)
N(02)#1-Ag-N(1')-C(2')	-38.2(4)
N(1")-Ag-N(1')-C(2')	174.1(4)
N(1)-Ag-N(1')-C(2')	11.5(3)
N(02)#1-Ag-N(1')-C(6')	136.5(3)
N(1")-Ag-N(1')-C(6')	-11.1(3)
N(1)-Ag-N(1')-C(6')	-173.7(3)
N(02)#1-Ag-N(1")-C(6")	26.6(5)
N(1')-Ag-N(1")-C(6")	179.4(4)
N(1)-Ag-N(1")-C(6")	-158.5(4)
N(02)#1-Ag-N(1")-C(2")	-140.5(3)
N(1')-Ag-N(1")-C(2")	12.3(3)
N(1)-Ag-N(1")-C(2")	34.4(4)
C(05)-N(01)-N(02)-C(03)	1.4(5)
C(02)-N(01)-N(02)-C(03)	169.5(4)
C(05)-N(01)-N(02)-Ag#1	-179.1(3)
C(02)-N(01)-N(02)-Ag#1	-11.0(5)
C(05')-N(01')-N(02')-C(03')	-0.8(5)
C(02)-N(01')-N(02')-C(03')	173.0(4)
C(05")-N(01")-N(02")-C(03")	0.7(5)
C(02)-N(01")-N(02")-C(03")	178.2(4)
C(6)-N(1)-C(2)-C(3)	-1.7(9)
Ag-N(1)-C(2)-C(3)	-166.8(5)
C(6)-N(1)-C(2)-C(2')	-179.7(5)
Ag-N(1)-C(2)-C(2')	15.2(6)
N(1)-C(2)-C(3)-C(4)	1.0(11)
C(2')-C(2)-C(3)-C(4)	178.9(7)
C(2)-C(3)-C(4)-C(5)	2.4(13)
C(3)-C(4)-C(5)-C(6)	-4.8(13)
C(4)-C(5)-C(6)-N(1)	4.3(12)
C(2)-N(1)-C(6)-C(5)	-1.0(11)
Ag-N(1)-C(6)-C(5)	162.2(6)
C(6')-N(1')-C(2')-C(3')	-2.7(6)
Ag-N(1')-C(2')-C(3')	172.1(3)
C(6')-N(1')-C(2')-C(2)	176.7(4)
Ag-N(1')-C(2')-C(2)	-8.5(5)
N(1)-C(2)-C(2')-N(1')	-5.9(6)
C(3)-C(2)-C(2')-N(1')	176.1(5)
N(1)-C(2)-C(2')-C(3')	173.5(5)
C(3)-C(2)-C(2')-C(3')	-4.5(8)
N(1')-C(2')-C(3')-C(4')	0.2(7)
C(2)-C(2')-C(3')-C(4')	-179.1(4)
C(2')-C(3')-C(4')-C(5')	1.3(6)
C(2')-C(3')-C(4')-C(1"')	-178.7(4)
C(3')-C(4')-C(5')-C(6')	-0.4(6)
C(1"')-C(4')-C(5')-C(6')	179.7(4)
C(2')-N(1')-C(6')-C(5')	3.7(6)
Ag-N(1')-C(6')-C(5')	-171.4(3)
C(2')-N(1')-C(6')-C(2")	-176.1(4)
Ag-N(1')-C(6')-C(2")	8.9(4)
C(4')-C(5')-C(6')-N(1')	-2.1(6)
C(4')-C(5')-C(6')-C(2")	177.6(4)
C(6")-N(1")-C(2")-C(3")	-0.3(6)

Ag-N(1")-C(2")-C(3")	167.8(3)
C(6")-N(1")-C(2")-C(6')	179.4(4)
Ag-N(1")-C(2")-C(6')	-12.6(5)
N(1')-C(6')-C(2")-N(1")	2.7(5)
C(5')-C(6')-C(2")-N(1")	-177.0(4)
N(1')-C(6')-C(2")-C(3")	-177.6(4)
C(5')-C(6')-C(2")-C(3")	2.6(6)
N(1")-C(2")-C(3")-C(4")	-1.1(7)
C(6')-C(2")-C(3")-C(4")	179.3(4)
C(2")-C(3")-C(4")-C(5")	1.4(8)
C(3")-C(4")-C(5")-C(6")	-0.4(8)
C(2")-N(1")-C(6")-C(5")	1.4(7)
Ag-N(1")-C(6")-C(5")	-165.5(4)
C(4")-C(5")-C(6")-N(1")	-1.0(8)
C(5')-C(4')-C(1"')-C(6"')	-170.7(4)
C(3')-C(4')-C(1"')-C(6"')	9.4(6)
C(5')-C(4')-C(1"')-C(2"')	8.7(6)
C(3')-C(4')-C(1"')-C(2"')	-171.2(4)
C(2"')-C(1"')-C(6"')-C(5"')	-1.4(7)
C(4')-C(1"')-C(6"')-C(5"')	178.1(4)
C(1"')-C(6"')-C(5"')-C(4"')	0.9(8)
C(6"')-C(5"')-C(4"')-C(3"')	0.4(7)
C(6"')-C(5"')-C(4"')-C(7)	-179.3(4)
C(5"')-C(4"')-C(3"')-C(2"')	-1.2(6)
C(7)-C(4"')-C(3"')-C(2"')	178.4(4)
C(4"')-C(3"')-C(2"')-C(1"')	0.8(6)
C(6"')-C(1"')-C(2"')-C(3"')	0.5(6)
C(4')-C(1"')-C(2"')-C(3"')	-178.9(4)
C(01)-O-C(7)-C(4"')	-63.4(5)
C(5"')-C(4"')-C(7)-O	134.4(4)
C(3"')-C(4"')-C(7)-O	-45.2(6)
C(7)-O-C(01)-C(02)	164.5(3)
C(05")-N(01")-C(02)-N(01')	-79.5(6)
N(02")-N(01")-C(02)-N(01')	103.5(4)
C(05")-N(01")-C(02)-N(01)	37.4(6)
N(02")-N(01")-C(02)-N(01)	-139.7(4)
C(05")-N(01")-C(02)-C(01)	156.5(4)
N(02")-N(01")-C(02)-C(01)	-20.5(5)
C(05')-N(01')-C(02)-N(01")	-133.1(5)
N(02')-N(01')-C(02)-N(01")	54.4(5)
C(05')-N(01')-C(02)-N(01)	110.1(5)
N(02')-N(01')-C(02)-N(01)	-62.4(5)
C(05')-N(01')-C(02)-C(01)	-9.9(6)
N(02')-N(01')-C(02)-C(01)	177.6(4)
C(05)-N(01)-C(02)-N(01")	-144.3(4)
N(02)-N(01)-C(02)-N(01")	49.7(5)
C(05)-N(01)-C(02)-N(01')	-25.4(6)
N(02)-N(01)-C(02)-N(01')	168.5(3)
C(05)-N(01)-C(02)-C(01)	96.1(5)
N(02)-N(01)-C(02)-C(01)	-70.0(4)
O-C(01)-C(02)-N(01")	-166.6(3)
O-C(01)-C(02)-N(01')	70.1(4)
O-C(01)-C(02)-N(01)	-48.7(4)
N(01)-N(02)-C(03)-C(04)	-1.0(5)
Ag#1-N(02)-C(03)-C(04)	179.4(3)
N(02)-C(03)-C(04)-C(05)	0.3(6)
N(02)-N(01)-C(05)-C(04)	-1.3(5)
C(02)-N(01)-C(05)-C(04)	-168.2(4)
C(03)-C(04)-C(05)-N(01)	0.6(6)
N(01')-N(02')-C(03')-C(04')	0.7(6)
N(02')-C(03')-C(04')-C(05')	-0.4(6)

N(02')-N(01')-C(05')-C(04')	0.6(5)
C(02)-N(01')-C(05')-C(04')	-172.2(4)
C(03')-C(04')-C(05')-N(01')	-0.1(5)
N(01")-N(02")-C(03")-C(04")	-0.8(6)
N(02")-C(03")-C(04")-C(05")	0.6(7)
N(02")-N(01")-C(05")-C(04")	-0.4(6)
C(02)-N(01")-C(05")-C(04")	-177.7(4)
C(03")-C(04")-C(05")-N(01")	-0.1(7)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y,-z+2

Table A2.87. Hydrogen bonds for $[Ag_2(pzt)_2](BF_4)_2$, **4.2** [Å and °.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

Table A2.88. Crystal data and structure refinement for $[Cu(pzt)(ONO_2)_2].CH_3CN$, **4.7.**

Identification code	**4.7**
Empirical formula	C35 H30 Cu N12 O7
Formula weight	794.25
Temperature	168(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, *P*-1
Unit cell dimensions	a = 8.8307(7) Å alpha = 88.3580(10)°. b = 11.8013(9) Å beta = 79.6760(10)°. c = 17.3963(13) Å gamma = 83.2590(10)°.
Volume	1771.2(2) $Å^3$
Z, Calculated density	2, 1.489 Mg/m3
Absorption coefficient	0.685 mm^{-1}
F(000)	818
Crystal size	0.85 x 0.45 x 0.10 mm
Theta range for data collection	2.10 to 25.05 °.
Limiting indices	-10<=h<=10, -14<=k<=6, -20<=l<=20
Reflections collected / unique	20709 / 6267 [R(int) = 0.0197]
Completeness to theta = 25.05	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9347 and 0.5937
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6267 / 0 / 509
Goodness-of-fit on F^2	1.083
Final R indices [I>2sigma(I)]	R1 = 0.0357, wR2 = 0.0990
R indices (all data)	R1 = 0.0420, wR2 = 0.1048
Largest diff. peak and hole	0.850 and -0.292 e.$Å^{-3}$

Table A2.89. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for [Cu(pzt)$(ONO_2)_2$].CH_3CN, **4.7.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cu	6284(1)	7017(1)	11246(1)	27(1)
N(1')	5305(2)	7347(1)	10345(1)	25(1)
N(1")	7204(2)	5630(2)	10573(1)	27(1)
N(1)	4973(2)	8500(2)	11590(1)	29(1)
N(4)	5701(2)	5618(2)	12519(1)	37(1)
O(41)	6617(2)	6388(1)	12281(1)	37(1)
O(42)	5894(3)	5067(2)	13105(1)	58(1)
O(43)	4696(2)	5471(2)	12130(1)	50(1)
N(5)	8926(2)	8385(2)	10736(1)	29(1)
O(51)	8519(2)	7654(1)	11264(1)	34(1)
O(53)	10196(2)	8757(1)	10703(1)	39(1)
O(52)	8050(2)	8702(2)	10276(1)	56(1)
N(01)	791(3)	7791(2)	3849(1)	43(1)
C(05)	1605(4)	7865(3)	3114(2)	52(1)
C(04)	556(4)	8009(3)	2626(2)	54(1)
C(03)	-863(4)	7993(3)	3111(2)	57(1)
N(02)	-734(3)	7863(2)	3857(1)	49(1)
N(01')	2885(3)	8013(2)	4477(1)	49(1)
C(05')	4318(4)	7478(3)	4477(2)	61(1)
C(04')	5287(4)	8303(4)	4403(2)	73(1)
C(03')	4342(4)	9322(4)	4358(2)	70(1)
N(02')	2854(3)	9170(2)	4407(2)	62(1)
N(01")	1703(3)	6311(2)	4668(1)	46(1)
C(05")	1654(4)	5688(3)	5330(2)	62(1)
C(04")	2230(4)	4597(3)	5122(2)	67(1)
C(03")	2613(4)	4624(3)	4320(2)	70(1)
N(02")	2302(4)	5660(3)	4027(2)	69(1)
C(2')	4303(2)	8298(2)	10338(1)	25(1)
C(3')	3582(2)	8565(2)	9705(1)	25(1)
C(4')	3895(2)	7834(2)	9058(1)	25(1)
C(5')	4962(2)	6854(2)	9082(1)	26(1)
C(6')	5639(2)	6639(2)	9733(1)	25(1)
C(2")	6762(2)	5640(2)	9868(1)	25(1)
C(3")	7306(2)	4774(2)	9331(1)	29(1)
C(4")	8338(2)	3877(2)	9527(1)	31(1)
C(5")	8791(3)	3869(2)	10249(1)	32(1)
C(6")	8201(2)	4753(2)	10757(1)	31(1)
C(2)	4125(2)	8974(2)	11063(1)	25(1)
C(3)	3219(3)	10015(2)	11190(1)	31(1)
C(4)	3211(3)	10599(2)	11874(1)	36(1)
C(5)	4091(3)	10120(2)	12407(1)	38(1)
C(6)	4942(3)	9067(2)	12254(1)	35(1)
C(1"')	3098(2)	8087(2)	8381(1)	28(1)
C(2"')	3685(3)	7593(2)	7649(1)	37(1)
C(3"')	2942(3)	7847(2)	7023(2)	44(1)
C(4"')	1585(3)	8600(2)	7100(2)	41(1)
C(5"')	1002(3)	9090(2)	7822(2)	39(1)
C(6"')	1724(3)	8837(2)	8456(1)	32(1)
C(7)	745(4)	8882(3)	6421(2)	59(1)
O	993(3)	7970(2)	5925(1)	67(1)
C(01)	275(3)	8094(3)	5261(2)	51(1)
C(02)	1403(3)	7539(2)	4568(2)	42(1)

C(9)	9109(5)	5952(3)	7534(3)	98(2)
C(8)	9976(6)	4881(4)	7310(3)	82(1)
N(6)	8790(12)	6714(7)	8011(8)	120(4)
N(6')	8142(10)	6751(7)	7533(6)	95(3)

Table A2.90. Selected bond lengths [Å] and angles [°] for $[Cu(pzt)(ONO_2)_2].CH_3CN$, **4.7.**

Cu–N(1')	1.9311(18)
Cu–O(41)	1.9847(16)
Cu–N(1)	2.0247(19)
Cu–N(1")	2.0410(18)
Cu–O(51)	2.1989(16)
N(1')–Cu–O(41)	159.30(7)
N(1')–Cu–N(1)	80.06(7)
O(41)–Cu–N(1)	99.52(7)
N(1')–Cu–N(1")	79.60(7)
O(41)–Cu–N(1")	99.10(7)
N(1)–Cu–N(1")	159.63(7)
N(1')–Cu–O(51)	118.74(7)
O(41)–Cu–O(51)	81.95(6)
N(1)–Cu–O(51)	95.48(7)
N(1")–Cu–O(51)	95.23(7)

Symmetry transformations used to generate equivalent atoms:

Table A2.91. Bond lengths [Å] and angles [°] for $[Cu(pzt)(ONO_2)_2].CH_3CN$, **4.7.**

Cu–N(1')	1.9311(18)
Cu–O(41)	1.9847(16)
Cu–N(1)	2.0247(19)
Cu–N(1")	2.0410(18)
Cu–O(51)	2.1989(16)
N(1')–C(6')	1.340(3)
N(1')–C(2')	1.347(3)
N(1")–C(6")	1.347(3)
N(1")–C(2")	1.353(3)
N(1)–C(6)	1.346(3)
N(1)–C(2)	1.353(3)
N(4)–O(42)	1.219(3)
N(4)–O(43)	1.239(3)
N(4)–O(41)	1.297(3)
N(5)–O(52)	1.231(3)
N(5)–O(53)	1.243(2)
N(5)–O(51)	1.275(2)
N(01)–N(02)	1.337(3)
N(01)–C(05)	1.356(4)
N(01)–C(02)	1.459(3)
C(05)–C(04)	1.359(4)
C(05)–H(05)	0.9300
C(04)–C(03)	1.382(5)
C(04)–H(04)	0.9300
C(03)–N(02)	1.324(4)
C(03)–H(03)	0.9300
N(01')–C(05')	1.347(4)
N(01')–N(02')	1.365(4)
N(01')–C(02)	1.463(3)
C(05')–C(04')	1.358(5)
C(05')–H(05')	0.9300
C(04')–C(03')	1.389(6)
C(04')–H(04')	0.9300
C(03')–N(02')	1.334(4)
C(03')–H(03')	0.9300
N(01")–C(05")	1.346(4)
N(01")–N(02")	1.363(4)
N(01")–C(02)	1.453(4)
C(05")–C(04")	1.362(5)
C(05")–H(05")	0.9300
C(04")–C(03")	1.376(5)
C(04")–H(04")	0.9300
C(03")–N(02")	1.329(4)
C(03")–H(03")	0.9300
C(2')–C(3')	1.379(3)
C(2')–C(2)	1.486(3)
C(3')–C(4')	1.406(3)
C(3')–H(3')	0.9300
C(4')–C(5')	1.407(3)
C(4')–C(1"')	1.484(3)
C(5')–C(6')	1.377(3)
C(5')–H(5')	0.9300
C(6')–C(2")	1.490(3)
C(2")–C(3")	1.388(3)
C(3")–C(4")	1.389(3)
C(3")–H(3")	0.9300
C(4")–C(5")	1.384(3)

C(4")-H(4")	0.9300
C(5")-C(6")	1.379(3)
C(5")-H(5")	0.9300
C(6")-H(6")	0.9300
C(2)-C(3)	1.386(3)
C(3)-C(4)	1.390(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.382(4)
C(4)-H(4)	0.9300
C(5)-C(6)	1.381(3)
C(5)-H(5)	0.9300
C(6)-H(6)	0.9300
C(1"')-C(2"')	1.402(3)
C(1"')-C(6"')	1.404(3)
C(2"')-C(3"')	1.377(3)
C(2"')-H(2"')	0.9300
C(3"')-C(4"')	1.394(4)
C(3"')-H(3"')	0.9300
C(4"')-C(5"')	1.386(4)
C(4"')-C(7)	1.512(3)
C(5"')-C(6"')	1.379(3)
C(5"')-H(5"')	0.9300
C(6"')-H(6"')	0.9300
C(7)-O	1.371(4)
C(7)-H(7A)	0.9700
C(7)-H(7B)	0.9700
O-C(01)	1.409(3)
C(01)-C(02)	1.529(4)
C(01)-H(01A)	0.9700
C(01)-H(01B)	0.9700
C(9)-N(6')	1.197(9)
C(9)-N(6)	1.215(12)
C(9)-C(8)	1.425(5)
C(8)-H(8A)	0.9600
C(8)-H(8B)	0.9600
C(8)-H(8C)	0.9600
N(6)-N(6')	1.089(13)

N(1')-Cu-O(41)	159.30(7)
N(1')-Cu-N(1)	80.06(7)
O(41)-Cu-N(1)	99.52(7)
N(1')-Cu-N(1")	79.60(7)
O(41)-Cu-N(1")	99.10(7)
N(1)-Cu-N(1")	159.63(7)
N(1')-Cu-O(51)	118.74(7)
O(41)-Cu-O(51)	81.95(6)
N(1)-Cu-O(51)	95.48(7)
N(1")-Cu-O(51)	95.23(7)
C(6')-N(1')-C(2')	120.92(18)
C(6')-N(1')-Cu	119.98(14)
C(2')-N(1')-Cu	119.10(14)
C(6")-N(1")-C(2")	118.81(19)
C(6")-N(1")-Cu	126.86(15)
C(2")-N(1")-Cu	114.31(13)
C(6)-N(1)-C(2)	118.67(19)
C(6)-N(1)-Cu	126.60(16)
C(2)-N(1)-Cu	114.62(14)
O(42)-N(4)-O(43)	123.7(2)
O(42)-N(4)-O(41)	118.3(2)
O(43)-N(4)-O(41)	117.94(19)
N(4)-O(41)-Cu	111.10(13)

O(52)-N(5)-O(53)	122.00(19)
O(52)-N(5)-O(51)	119.09(19)
O(53)-N(5)-O(51)	118.91(18)
N(5)-O(51)-Cu	115.78(13)
N(02)-N(01)-C(05)	111.9(2)
N(02)-N(01)-C(02)	120.3(2)
C(05)-N(01)-C(02)	127.5(2)
N(01)-C(05)-C(04)	106.8(3)
N(01)-C(05)-H(05)	126.6
C(04)-C(05)-H(05)	126.6
C(05)-C(04)-C(03)	104.6(3)
C(05)-C(04)-H(04)	127.7
C(03)-C(04)-H(04)	127.7
N(02)-C(03)-C(04)	112.5(3)
N(02)-C(03)-H(03)	123.8
C(04)-C(03)-H(03)	123.8
C(03)-N(02)-N(01)	104.2(2)
C(05')-N(01')-N(02')	113.0(3)
C(05')-N(01')-C(02)	129.6(3)
N(02')-N(01')-C(02)	117.4(2)
N(01')-C(05')-C(04')	106.6(3)
N(01')-C(05')-H(05')	126.7
C(04')-C(05')-H(05')	126.7
C(05')-C(04')-C(03')	105.1(3)
C(05')-C(04')-H(04')	127.4
C(03')-C(04')-H(04')	127.4
N(02')-C(03')-C(04')	112.7(3)
N(02')-C(03')-H(03')	123.7
C(04')-C(03')-H(03')	123.7
C(03')-N(02')-N(01')	102.6(3)
C(05")-N(01")-N(02")	111.4(3)
C(05")-N(01")-C(02)	129.4(3)
N(02")-N(01")-C(02)	118.7(2)
N(01")-C(05")-C(04")	107.2(3)
N(01")-C(05")-H(05")	126.4
C(04")-C(05")-H(05")	126.4
C(05")-C(04")-C(03")	105.2(3)
C(05")-C(04")-H(04")	127.4
C(03")-C(04")-H(04")	127.4
N(02")-C(03")-C(04")	112.3(3)
N(02")-C(03")-H(03")	123.9
C(04")-C(03")-H(03")	123.9
C(03")-N(02")-N(01")	104.0(3)
N(1')-C(2')-C(3')	120.87(19)
N(1')-C(2')-C(2)	112.25(18)
C(3')-C(2')-C(2)	126.88(19)
C(2')-C(3')-C(4')	119.58(19)
C(2')-C(3')-H(3')	120.2
C(4')-C(3')-H(3')	120.2
C(3')-C(4')-C(5')	117.96(19)
C(3')-C(4')-C(1"')	120.47(19)
C(5')-C(4')-C(1"')	121.55(19)
C(6')-C(5')-C(4')	119.41(19)
C(6')-C(5')-H(5')	120.3
C(4')-C(5')-H(5')	120.3
N(1')-C(6')-C(5')	121.25(18)
N(1')-C(6')-C(2")	111.88(18)
C(5')-C(6')-C(2")	126.86(19)
N(1")-C(2")-C(3")	121.81(19)
N(1")-C(2")-C(6')	114.17(18)
C(3")-C(2")-C(6')	124.02(19)

C(2")-C(3")-C(4")	118.7(2)
C(2")-C(3")-H(3")	120.6
C(4")-C(3")-H(3")	120.6
C(5")-C(4")-C(3")	119.4(2)
C(5")-C(4")-H(4")	120.3
C(3")-C(4")-H(4")	120.3
C(6")-C(5")-C(4")	118.9(2)
C(6")-C(5")-H(5")	120.5
C(4")-C(5")-H(5")	120.5
N(1")-C(6")-C(5")	122.3(2)
N(1")-C(6")-H(6")	118.9
C(5")-C(6")-H(6")	118.9
N(1)-C(2)-C(3)	122.1(2)
N(1)-C(2)-C(2')	113.96(18)
C(3)-C(2)-C(2')	123.9(2)
C(2)-C(3)-C(4)	118.7(2)
C(2)-C(3)-H(3)	120.7
C(4)-C(3)-H(3)	120.7
C(5)-C(4)-C(3)	119.1(2)
C(5)-C(4)-H(4)	120.4
C(3)-C(4)-H(4)	120.4
C(6)-C(5)-C(4)	119.3(2)
C(6)-C(5)-H(5)	120.3
C(4)-C(5)-H(5)	120.3
N(1)-C(6)-C(5)	122.0(2)
N(1)-C(6)-H(6)	119.0
C(5)-C(6)-H(6)	119.0
C(2"')-C(1"')-C(6"')	117.9(2)
C(2"')-C(1"')-C(4')	121.55(19)
C(6"')-C(1"')-C(4')	120.6(2)
C(3"')-C(2"')-C(1"')	120.9(2)
C(3"')-C(2"')-H(2"')	119.5
C(1"')-C(2"')-H(2"')	119.5
C(2"')-C(3"')-C(4"')	120.9(2)
C(2"')-C(3"')-H(3"')	119.5
C(4"')-C(3"')-H(3"')	119.5
C(5"')-C(4"')-C(3"')	118.3(2)
C(5"')-C(4"')-C(7)	120.0(2)
C(3"')-C(4"')-C(7)	121.7(2)
C(6"')-C(5"')-C(4"')	121.4(2)
C(6"')-C(5"')-H(5"')	119.3
C(4"')-C(5"')-H(5"')	119.3
C(5"')-C(6"')-C(1"')	120.5(2)
C(5"')-C(6"')-H(6"')	119.7
C(1"')-C(6"')-H(6"')	119.7
O-C(7)-C(4"')	109.7(2)
O-C(7)-H(7A)	109.7
C(4"')-C(7)-H(7A)	109.7
O-C(7)-H(7B)	109.7
C(4"')-C(7)-H(7B)	109.7
H(7A)-C(7)-H(7B)	108.2
C(7)-O-C(01)	116.7(2)
O-C(01)-C(02)	108.3(2)
O-C(01)-H(01A)	110.0
C(02)-C(01)-H(01A)	110.0
O-C(01)-H(01B)	110.0
C(02)-C(01)-H(01B)	110.0
H(01A)-C(01)-H(01B)	108.4
N(01")-C(02)-N(01)	109.6(2)
N(01")-C(02)-N(01')	107.9(2)
N(01)-C(02)-N(01')	107.3(2)

N(01")-C(02)-C(01)	112.0(2)
N(01)-C(02)-C(01)	109.8(2)
N(01')-C(02)-C(01)	110.0(2)
N(6')-C(9)-N(6)	53.7(6)
N(6')-C(9)-C(8)	158.4(8)
N(6)-C(9)-C(8)	147.5(8)
C(9)-C(8)-H(8A)	109.5
C(9)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(9)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
N(6')-N(6)-C(9)	62.3(10)
N(6)-N(6')-C(9)	64.0(8)

Symmetry transformations used to generate equivalent atoms:

Table A2.92. Anisotropic displacement parameters (A^2 x 10^3) for $[Cu(pzt)(ONO_2)_2].CH_3CN$, **4.7.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Cu	28(1)	28(1)	26(1)	-2(1)	-10(1)	2(1)
N(1')	24(1)	23(1)	27(1)	-2(1)	-5(1)	1(1)
N(1")	25(1)	26(1)	31(1)	1(1)	-8(1)	1(1)
N(1)	28(1)	33(1)	27(1)	-3(1)	-7(1)	-2(1)
N(4)	43(1)	34(1)	30(1)	-3(1)	-2(1)	0(1)
O(41)	44(1)	38(1)	31(1)	6(1)	-13(1)	-9(1)
O(42)	74(1)	53(1)	44(1)	19(1)	-8(1)	-8(1)
O(43)	47(1)	63(1)	45(1)	-7(1)	-9(1)	-19(1)
N(5)	30(1)	22(1)	35(1)	-1(1)	-6(1)	2(1)
O(51)	31(1)	38(1)	34(1)	7(1)	-9(1)	-5(1)
O(53)	30(1)	31(1)	55(1)	3(1)	-5(1)	-4(1)
O(52)	53(1)	62(1)	61(1)	32(1)	-32(1)	-15(1)
N(01)	46(1)	51(1)	36(1)	6(1)	-17(1)	-11(1)
C(05)	56(2)	66(2)	38(2)	8(1)	-11(1)	-20(2)
C(04)	75(2)	58(2)	34(1)	5(1)	-22(1)	-16(2)
C(03)	63(2)	65(2)	54(2)	4(2)	-37(2)	-6(2)
N(02)	41(1)	66(2)	42(1)	1(1)	-18(1)	1(1)
N(01')	49(1)	57(2)	47(1)	5(1)	-21(1)	-14(1)
C(05')	44(2)	78(2)	64(2)	3(2)	-18(1)	-6(2)
C(04')	51(2)	104(3)	70(2)	1(2)	-20(2)	-24(2)
C(03')	82(2)	88(3)	53(2)	9(2)	-28(2)	-44(2)
N(02')	74(2)	62(2)	62(2)	8(1)	-32(1)	-24(1)
N(01")	46(1)	51(1)	43(1)	5(1)	-14(1)	-4(1)
C(05")	73(2)	60(2)	51(2)	15(2)	-9(2)	-5(2)
C(04")	76(2)	53(2)	74(2)	19(2)	-18(2)	-5(2)
C(03")	74(2)	55(2)	78(2)	-2(2)	-15(2)	2(2)
N(02")	87(2)	61(2)	56(2)	-5(1)	-13(2)	3(2)
C(2')	22(1)	23(1)	28(1)	0(1)	-3(1)	-2(1)
C(3')	23(1)	23(1)	29(1)	1(1)	-4(1)	1(1)
C(4')	23(1)	26(1)	28(1)	2(1)	-4(1)	-3(1)
C(5')	25(1)	25(1)	26(1)	-3(1)	-4(1)	0(1)
C(6')	22(1)	24(1)	28(1)	-1(1)	-3(1)	-1(1)
C(2")	21(1)	24(1)	29(1)	1(1)	-4(1)	-1(1)
C(3")	26(1)	30(1)	30(1)	-2(1)	-5(1)	1(1)
C(4")	27(1)	25(1)	38(1)	-3(1)	-2(1)	3(1)
C(5")	25(1)	27(1)	42(1)	4(1)	-5(1)	2(1)
C(6")	27(1)	33(1)	33(1)	4(1)	-10(1)	0(1)
C(2)	24(1)	26(1)	26(1)	-2(1)	-3(1)	-4(1)
C(3)	33(1)	28(1)	31(1)	0(1)	-4(1)	-1(1)
C(4)	43(1)	26(1)	37(1)	-5(1)	-1(1)	0(1)
C(5)	45(1)	38(1)	31(1)	-11(1)	-5(1)	-6(1)
C(6)	38(1)	39(1)	30(1)	-5(1)	-10(1)	-3(1)
C(1"')	28(1)	26(1)	29(1)	1(1)	-7(1)	-2(1)
C(2"')	37(1)	40(1)	32(1)	-4(1)	-10(1)	10(1)
C(3"')	50(2)	49(2)	30(1)	-8(1)	-12(1)	11(1)
C(4"')	46(1)	43(1)	36(1)	-1(1)	-18(1)	6(1)
C(5"')	38(1)	37(1)	42(1)	-2(1)	-14(1)	10(1)
C(6"')	32(1)	32(1)	32(1)	-3(1)	-8(1)	4(1)
C(7)	70(2)	62(2)	46(2)	-12(1)	-32(2)	23(2)
O	95(2)	66(1)	44(1)	-6(1)	-40(1)	19(1)
C(01)	55(2)	62(2)	38(1)	3(1)	-19(1)	4(1)
C(02)	43(1)	50(2)	38(1)	5(1)	-18(1)	-7(1)
C(9)	88(3)	48(2)	135(4)	-2(2)	40(3)	-5(2)

C(8)	98(3)	79(3)	65(2)	−21(2)	−18(2)	13(2)
N(6)	98(7)	46(4)	189(12)	15(6)	37(7)	−5(4)
N(6')	70(5)	59(4)	146(9)	−38(5)	5(5)	7(4)

Table A2.93. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for [Cu(pzt)$(ONO_2)_2$].CH_3CN, **4.7.**

	x	y	z	U(eq)
H(05)	2677	7825	2971	63
H(04)	749	8098	2085	65
H(03)	-1804	8066	2935	69
H(05')	4591	6696	4519	73
H(04')	6353	8207	4386	87
H(03')	4706	10033	4301	84
H(05")	1295	5953	5836	74
H(04")	2341	3969	5451	81
H(03")	3041	3989	4017	84
H(3')	2893	9225	9706	30
H(5')	5207	6356	8661	31
H(3")	6986	4793	8849	35
H(4")	8721	3288	9176	37
H(5")	9482	3276	10389	38
H(6")	8501	4744	11243	37
H(3)	2628	10317	10825	37
H(4)	2621	11303	11971	44
H(5)	4110	10503	12864	46
H(6)	5511	8740	12621	42
H(2"')	4589	7088	7585	44
H(3"')	3351	7510	6542	52
H(5"')	104	9601	7881	47
H(6"')	1298	9166	8938	38
H(7A)	-356	9058	6614	71
H(7B)	1120	9546	6142	71
H(01A)	10	8897	5152	61
H(01B)	-670	7729	5353	61
H(8A)	9486	4515	6950	170(30)
H(8B)	10017	4406	7765	210(30)
H(8C)	11009	5000	7064	200(30)

Table A2.94. Selected torsion angles [°] for [Cu(pzt)$(ONO_2)_2$].CH_3CN, **4.7.**

C(3')-C(4')-C(1"')-C(2"')	160.9(2)
C(5')-C(4')-C(1"')-C(2"')	-20.2(3)
C(3')-C(4')-C(1"')-C(6"')	-18.9(3)
C(5')-C(4')-C(1"')-C(6"')	160.1(2)

Symmetry transformations used to generate equivalent atoms:

Table A2.95. Torsion angles [°] for [Cu(pzt)$(ONO_2)_2$].CH_3CN, **4.7.**

O(41)-Cu-N(1')-C(6')	-90.1(2)
N(1)-Cu-N(1')-C(6')	179.22(17)
N(1")-Cu-N(1')-C(6')	-1.89(15)
O(51)-Cu-N(1')-C(6')	88.39(16)
O(41)-Cu-N(1')-C(2')	90.6(2)
N(1)-Cu-N(1')-C(2')	-0.04(15)
N(1")-Cu-N(1')-C(2')	178.85(16)
O(51)-Cu-N(1')-C(2')	-90.87(16)
N(1')-Cu-N(1")-C(6")	-179.24(19)
O(41)-Cu-N(1")-C(6")	-20.20(19)
N(1)-Cu-N(1")-C(6")	-176.1(2)
O(51)-Cu-N(1")-C(6")	62.45(19)
N(1')-Cu-N(1")-C(2")	2.28(14)
O(41)-Cu-N(1")-C(2")	161.32(15)
N(1)-Cu-N(1")-C(2")	5.4(3)
O(51)-Cu-N(1")-C(2")	-116.03(15)
N(1')-Cu-N(1)-C(6)	-176.6(2)
O(41)-Cu-N(1)-C(6)	24.4(2)
N(1")-Cu-N(1)-C(6)	-179.7(2)
O(51)-Cu-N(1)-C(6)	-58.3(2)
N(1')-Cu-N(1)-C(2)	-0.59(15)
O(41)-Cu-N(1)-C(2)	-159.59(15)
N(1")-Cu-N(1)-C(2)	-3.7(3)
O(51)-Cu-N(1)-C(2)	117.68(15)
O(42)-N(4)-O(41)-Cu	171.97(17)
O(43)-N(4)-O(41)-Cu	-6.8(2)
N(1')-Cu-O(41)-N(4)	13.9(3)
N(1)-Cu-O(41)-N(4)	100.97(15)
N(1")-Cu-O(41)-N(4)	-70.74(15)
O(51)-Cu-O(41)-N(4)	-164.78(15)
O(52)-N(5)-O(51)-Cu	1.9(3)
O(53)-N(5)-O(51)-Cu	-178.19(14)
N(1')-Cu-O(51)-N(5)	12.00(17)
O(41)-Cu-O(51)-N(5)	-168.54(15)
N(1)-Cu-O(51)-N(5)	-69.66(15)
N(1")-Cu-O(51)-N(5)	92.99(15)
N(02)-N(01)-C(05)-C(04)	1.0(3)
C(02)-N(01)-C(05)-C(04)	174.4(3)
N(01)-C(05)-C(04)-C(03)	-1.0(3)
C(05)-C(04)-C(03)-N(02)	0.7(4)
C(04)-C(03)-N(02)-N(01)	-0.1(4)
C(05)-N(01)-N(02)-C(03)	-0.5(3)
C(02)-N(01)-N(02)-C(03)	-174.5(2)
N(02')-N(01')-C(05')-C(04')	0.0(4)

C(02)-N(01')-C(05')-C(04')	-177.7(3)
N(01')-C(05')-C(04')-C(03')	-0.3(4)
C(05')-C(04')-C(03')-N(02')	0.5(4)
C(04')-C(03')-N(02')-N(01')	-0.5(4)
C(05')-N(01')-N(02')-C(03')	0.3(3)
C(02)-N(01')-N(02')-C(03')	178.3(2)
N(02")-N(01")-C(05")-C(04")	-0.2(4)
C(02)-N(01")-C(05")-C(04")	-171.7(3)
N(01")-C(05")-C(04")-C(03")	0.3(4)
C(05")-C(04")-C(03")-N(02")	-0.2(5)
C(04")-C(03")-N(02")-N(01")	0.1(4)
C(05")-N(01")-N(02")-C(03")	0.1(4)
C(02)-N(01")-N(02")-C(03")	172.6(3)
C(6')-N(1')-C(2')-C(3')	0.6(3)
Cu-N(1')-C(2')-C(3')	179.89(15)
C(6')-N(1')-C(2')-C(2)	-178.66(18)
Cu-N(1')-C(2')-C(2)	0.6(2)
N(1')-C(2')-C(3')-C(4')	0.1(3)
C(2)-C(2')-C(3')-C(4')	179.26(19)
C(2')-C(3')-C(4')-C(5')	-0.8(3)
C(2')-C(3')-C(4')-C(1"')	178.19(19)
C(3')-C(4')-C(5')-C(6')	0.8(3)
C(1"')-C(4')-C(5')-C(6')	-178.15(19)
C(2')-N(1')-C(6')-C(5')	-0.6(3)
Cu-N(1')-C(6')-C(5')	-179.85(15)
C(2')-N(1')-C(6')-C(2")	-179.59(18)
Cu-N(1')-C(6')-C(2")	1.2(2)
C(4')-C(5')-C(6')-N(1')	-0.1(3)
C(4')-C(5')-C(6')-C(2")	178.69(19)
C(6")-N(1")-C(2")-C(3")	0.0(3)
Cu-N(1")-C(2")-C(3")	178.64(16)
C(6")-N(1")-C(2")-C(6')	179.07(18)
Cu-N(1")-C(2")-C(6')	-2.3(2)
N(1')-C(6')-C(2")-N(1")	0.9(3)
C(5')-C(6')-C(2")-N(1")	-178.1(2)
N(1')-C(6')-C(2")-C(3")	179.87(19)
C(5')-C(6')-C(2")-C(3")	1.0(3)
N(1")-C(2")-C(3")-C(4")	-0.3(3)
C(6')-C(2")-C(3")-C(4")	-179.2(2)
C(2")-C(3")-C(4")-C(5")	0.2(3)
C(3")-C(4")-C(5")-C(6")	0.1(3)
C(2")-N(1")-C(6")-C(5")	0.3(3)
Cu-N(1")-C(6")-C(5")	-178.11(16)
C(4")-C(5")-C(6")-N(1")	-0.4(3)
C(6)-N(1)-C(2)-C(3)	-0.7(3)
Cu-N(1)-C(2)-C(3)	-177.02(17)
C(6)-N(1)-C(2)-C(2')	177.39(19)
Cu-N(1)-C(2)-C(2')	1.0(2)
N(1')-C(2')-C(2)-N(1)	-1.1(3)
C(3')-C(2')-C(2)-N(1)	179.7(2)
N(1')-C(2')-C(2)-C(3)	177.0(2)
C(3')-C(2')-C(2)-C(3)	-2.3(3)
N(1)-C(2)-C(3)-C(4)	1.4(3)
C(2')-C(2)-C(3)-C(4)	-176.4(2)
C(2)-C(3)-C(4)-C(5)	-0.7(3)
C(3)-C(4)-C(5)-C(6)	-0.8(4)
C(2)-N(1)-C(6)-C(5)	-0.9(3)
Cu-N(1)-C(6)-C(5)	174.98(18)
C(4)-C(5)-C(6)-N(1)	1.6(4)
C(3')-C(4')-C(1"')-C(2"')	160.9(2)
C(5')-C(4')-C(1"')-C(2"')	-20.2(3)

C(3')-C(4')-C(1"')-C(6"')	-18.9(3)
C(5')-C(4')-C(1"')-C(6"')	160.1(2)
C(6"')-C(1"')-C(2"')-C(3"')	0.6(4)
C(4')-C(1"')-C(2"')-C(3"')	-179.2(2)
C(1"')-C(2"')-C(3"')-C(4"')	0.0(4)
C(2"')-C(3"')-C(4"')-C(5"')	0.1(4)
C(2"')-C(3"')-C(4"')-C(7)	-179.5(3)
C(3"')-C(4"')-C(5"')-C(6"')	-0.7(4)
C(7)-C(4"')-C(5"')-C(6"')	178.9(3)
C(4"')-C(5"')-C(6"')-C(1"')	1.3(4)
C(2"')-C(1"')-C(6"')-C(5"')	-1.2(3)
C(4')-C(1"')-C(6"')-C(5"')	178.6(2)
C(5"')-C(4"')-C(7)-O	-150.2(3)
C(3"')-C(4"')-C(7)-O	29.4(4)
C(4"')-C(7)-O-C(01)	179.6(3)
C(7)-O-C(01)-C(02)	140.7(3)
C(05")-N(01")-C(02)-N(01)	-150.0(3)
N(02")-N(01")-C(02)-N(01)	39.0(3)
C(05")-N(01")-C(02)-N(01')	93.4(3)
N(02")-N(01")-C(02)-N(01')	-77.5(3)
C(05")-N(01")-C(02)-C(01)	-27.8(4)
N(02")-N(01")-C(02)-C(01)	161.2(3)
N(02)-N(01)-C(02)-N(01")	88.4(3)
C(05)-N(01)-C(02)-N(01")	-84.5(3)
N(02)-N(01)-C(02)-N(01')	-154.6(2)
C(05)-N(01)-C(02)-N(01')	32.4(4)
N(02)-N(01)-C(02)-C(01)	-35.1(3)
C(05)-N(01)-C(02)-C(01)	152.0(3)
C(05')-N(01')-C(02)-N(01")	0.1(4)
N(02')-N(01')-C(02)-N(01")	-177.4(2)
C(05')-N(01')-C(02)-N(01)	-117.9(3)
N(02')-N(01')-C(02)-N(01)	64.5(3)
C(05')-N(01')-C(02)-C(01)	122.6(3)
N(02')-N(01')-C(02)-C(01)	-54.9(3)
O-C(01)-C(02)-N(01")	65.9(3)
O-C(01)-C(02)-N(01)	-172.0(2)
O-C(01)-C(02)-N(01')	-54.1(3)
C(8)-C(9)-N(6)-N(6')	-173.7(10)
C(8)-C(9)-N(6')-N(6)	170.8(14)

Symmetry transformations used to generate equivalent atoms:

Table A2.96. Hydrogen bonds for [Cu(pzt)(ONO_2)$_2$].CH_3CN, **4.7** [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

Table A2.97. Crystal data and structure refinement for [Zn(pzt)Cl_2], **4.8.**

Identification code	**4.8**
Empirical formula	C35 H30 Cl2 N10 O Zn
Formula weight	742.96
Temperature	93(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, *P* -1
Unit cell dimensions	a = 8.3507(15) Å alpha = 86.826(3)°. b = 11.1461(15) Å beta = 82.396(2)°. c = 18.124(3) Å gamma = 85.480(2)°.
Volume	1665.2(5) $Å^3$
Z, Calculated density	2, 1.482 Mg/m^3
Absorption coefficient	0.945 mm^{-1}
F(000)	764
Crystal size	0.88 x 0.30 x 0.10 mm
Theta range for data collection	2.20 to 26.47°.
Limiting indices	-9<=h<=10, -13<=k<=13, -20<=l<=22
Reflections collected / unique	14016 / 6541 [R(int) = 0.0254]
Completeness to theta = 25.00	96.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9114 and 0.4902
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6541 / 0 / 443
Goodness-of-fit on F^2	1.019
Final R indices [I>2sigma(I)]	R1 = 0.0360, wR2 = 0.0869
R indices (all data)	R1 = 0.0509, wR2 = 0.0957
Largest diff. peak and hole	0.703 and -0.346 $eÅ^{-3}$

Table A2.98. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for [Zn(pzt)Cl_2], **4.8.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Zn	6340(1)	7273(1)	6101(1)	18(1)
N(1)	7197(2)	5694(2)	5462(1)	18(1)
N(1')	5182(2)	7472(2)	5153(1)	17(1)
N(1")	4574(2)	8806(2)	6296(1)	19(1)
C(2)	6748(3)	5700(2)	4782(1)	18(1)
C(3)	7279(3)	4793(2)	4297(1)	22(1)
C(4)	8315(3)	3857(2)	4532(2)	24(1)
C(5)	8765(3)	3849(2)	5234(2)	23(1)
C(6)	8183(3)	4784(2)	5686(1)	21(1)
C(2')	5597(3)	6723(2)	4599(1)	17(1)
C(3')	4970(3)	6897(2)	3936(1)	19(1)
C(4')	3875(3)	7880(2)	3824(1)	19(1)
C(5')	3463(3)	8649(2)	4411(1)	19(1)
C(6')	4118(3)	8416(2)	5066(1)	18(1)
C(2")	3742(3)	9160(2)	5730(1)	18(1)
C(3")	2620(3)	10143(2)	5768(1)	21(1)
C(4")	2387(3)	10784(2)	6409(1)	25(1)
C(5")	3240(3)	10425(2)	6989(1)	25(1)
C(6")	4320(3)	9432(2)	6914(1)	22(1)
C(1"')	3159(3)	8083(2)	3122(1)	20(1)
C(2"')	1953(3)	8986(2)	3043(2)	30(1)
C(3"')	1258(3)	9168(2)	2401(2)	31(1)
C(4"')	1720(3)	8456(2)	1809(1)	24(1)
C(5"')	2918(4)	7550(3)	1876(2)	41(1)
C(6"')	3623(4)	7360(3)	2521(2)	40(1)
C(7)	845(3)	8636(3)	1140(2)	31(1)
O	1744(2)	8088(2)	536(1)	36(1)
C(01)	810(3)	8025(3)	-48(1)	31(1)
C(02)	1930(3)	7522(2)	-710(1)	29(1)
N(01)	2459(3)	6269(2)	-567(1)	34(1)
N(02)	1782(3)	5651(2)	44(1)	36(1)
N(01')	1075(3)	7635(2)	-1361(1)	26(1)
N(02')	-563(3)	7712(2)	-1275(1)	31(1)
N(01")	3346(3)	8207(2)	-870(1)	32(1)
N(02")	3123(3)	9379(2)	-1107(2)	40(1)
C(03)	2387(4)	4536(3)	-44(2)	41(1)
C(04)	3413(4)	4410(3)	-701(2)	50(1)
C(05)	3441(5)	5538(3)	-1024(2)	52(1)
C(03')	-924(4)	7738(3)	-1967(2)	35(1)
C(04')	464(4)	7666(3)	-2489(2)	36(1)
C(05')	1721(4)	7602(2)	-2083(2)	30(1)
C(03")	4521(4)	9834(4)	-1072(2)	49(1)
C(04")	5629(4)	8991(4)	-801(2)	58(1)
C(05")	4851(4)	7964(4)	-666(2)	50(1)
Cl(1)	8600(1)	8311(1)	5877(1)	21(1)
Cl(2)	5789(1)	6247(1)	7198(1)	32(1)
N	8295(5)	6613(3)	2692(3)	100(2)
C	9161(5)	6069(3)	2312(2)	51(1)
C'	10268(5)	5347(3)	1807(2)	53(1)

Table A2.99. Selected bond lengths [Å] and angles [°] for $[Zn(pzt)Cl_2]$, **4.8.**

Zn–N(1')	2.074(2)
Zn–N(1")	2.180(2)
Zn–N(1)	2.1818(19)
Zn–Cl(2)	2.2499(7)
Zn–Cl(1)	2.2704(7)
N(1')–Zn–N(1")	74.97(7)
N(1')–Zn–N(1)	74.71(7)
N(1")–Zn–N(1)	148.54(8)
N(1')–Zn–Cl(2)	132.68(6)
N(1")–Zn–Cl(2)	98.73(5)
N(1)–Zn–Cl(2)	95.63(5)
N(1')–Zn–Cl(1)	107.55(5)
N(1")–Zn–Cl(1)	97.90(5)
N(1)–Zn–Cl(1)	98.99(5)
Cl(2)–Zn–Cl(1)	119.75(3)

Symmetry transformations used to generate equivalent atoms:

Table A2.100. Bond lengths [Å] and angles [°] for [Zn(pzt)Cl_2], **4.8.**

Zn–N(1')	2.074(2)
Zn–N(1")	2.180(2)
Zn–N(1)	2.1818(19)
Zn–Cl(2)	2.2499(7)
Zn–Cl(1)	2.2704(7)
N(1)–C(2)	1.334(3)
N(1)–C(6)	1.335(3)
N(1')–C(2')	1.334(3)
N(1')–C(6')	1.339(3)
N(1")–C(6")	1.337(3)
N(1")–C(2")	1.339(3)
C(2)–C(3)	1.384(3)
C(2)–C(2')	1.485(3)
C(3)–C(4)	1.388(3)
C(3)–H(3)	0.9500
C(4)–C(5)	1.372(4)
C(4)–H(4)	0.9500
C(5)–C(6)	1.380(3)
C(5)–H(5)	0.9500
C(6)–H(6)	0.9500
C(2')–C(3')	1.371(3)
C(3')–C(4')	1.394(3)
C(3')–H(3')	0.9500
C(4')–C(5')	1.395(3)
C(4')–C(1"')	1.474(3)
C(5')–C(6')	1.374(3)
C(5')–H(5')	0.9500
C(6')–C(2")	1.484(3)
C(2")–C(3")	1.384(3)
C(3")–C(4")	1.380(3)
C(3")–H(3")	0.9500
C(4")–C(5")	1.371(4)
C(4")–H(4")	0.9500
C(5")–C(6")	1.373(3)
C(5")–H(5")	0.9500
C(6")–H(6")	0.9500
C(1"')–C(2"')	1.382(3)
C(1"')–C(6"')	1.386(4)
C(2"')–C(3"')	1.368(4)
C(2"')–H(2"')	0.9500
C(3"')–C(4"')	1.366(4)
C(3"')–H(3"')	0.9500
C(4"')–C(5"')	1.376(4)
C(4"')–C(7)	1.494(4)
C(5"')–C(6"')	1.375(4)
C(5"')–H(5"')	0.9500
C(6"')–H(6"')	0.9500
C(7)–O	1.385(3)
C(7)–H(7A)	0.9900
C(7)–H(7B)	0.9900
O–C(01)	1.403(3)
C(01)–C(02)	1.523(4)
C(01)–H(01A)	0.9900
C(01)–H(01B)	0.9900
C(02)–N(01")	1.447(4)
C(02)–N(01)	1.452(4)
C(02)–N(01')	1.453(3)
N(01)–C(05)	1.346(4)

N(01)-N(02)	1.353(3)
N(02)-C(03)	1.315(4)
N(01')-C(05')	1.348(3)
N(01')-N(02')	1.353(3)
N(02')-C(03')	1.325(4)
N(01")-C(05")	1.359(4)
N(01")-N(02")	1.359(4)
N(02")-C(03")	1.319(4)
C(03)-C(04)	1.379(4)
C(03)-H(03)	0.9500
C(04)-C(05)	1.358(5)
C(04)-H(04)	0.9500
C(05)-H(05)	0.9500
C(03')-C(04')	1.395(4)
C(03')-H(03')	0.9500
C(04')-C(05')	1.355(4)
C(04')-H(04')	0.9500
C(05')-H(05')	0.9500
C(03")-C(04")	1.383(5)
C(03")-H(03")	0.9500
C(04")-C(05")	1.356(6)
C(04")-H(04")	0.9500
C(05")-H(05")	0.9500
N-C	1.101(4)
C-C'	1.442(4)
C'-H'1	0.9800
C'-H'2	0.9800
C'-H'3	0.9800

N(1')-Zn-N(1")	74.97(7)
N(1')-Zn-N(1)	74.71(7)
N(1")-Zn-N(1)	148.54(8)
N(1')-Zn-Cl(2)	132.68(6)
N(1")-Zn-Cl(2)	98.73(5)
N(1)-Zn-Cl(2)	95.63(5)
N(1')-Zn-Cl(1)	107.55(5)
N(1")-Zn-Cl(1)	97.90(5)
N(1)-Zn-Cl(1)	98.99(5)
Cl(2)-Zn-Cl(1)	119.75(3)
C(2)-N(1)-C(6)	119.3(2)
C(2)-N(1)-Zn	115.65(15)
C(6)-N(1)-Zn	124.95(17)
C(2')-N(1')-C(6')	119.6(2)
C(2')-N(1')-Zn	120.18(15)
C(6')-N(1')-Zn	119.97(15)
C(6")-N(1")-C(2")	118.8(2)
C(6")-N(1")-Zn	125.26(16)
C(2")-N(1")-Zn	115.88(15)
N(1)-C(2)-C(3)	122.1(2)
N(1)-C(2)-C(2')	115.0(2)
C(3)-C(2)-C(2')	122.9(2)
C(2)-C(3)-C(4)	118.2(2)
C(2)-C(3)-H(3)	120.9
C(4)-C(3)-H(3)	120.9
C(5)-C(4)-C(3)	119.5(2)
C(5)-C(4)-H(4)	120.2
C(3)-C(4)-H(4)	120.2
C(4)-C(5)-C(6)	118.9(2)
C(4)-C(5)-H(5)	120.6
C(6)-C(5)-H(5)	120.6
N(1)-C(6)-C(5)	122.0(2)

N(1)-C(6)-H(6)	119.0
C(5)-C(6)-H(6)	119.0
N(1')-C(2')-C(3')	121.7(2)
N(1')-C(2')-C(2)	113.6(2)
C(3')-C(2')-C(2)	124.7(2)
C(2')-C(3')-C(4')	120.2(2)
C(2')-C(3')-H(3')	119.9
C(4')-C(3')-H(3')	119.9
C(5')-C(4')-C(3')	117.0(2)
C(5')-C(4')-C(1"')	121.5(2)
C(3')-C(4')-C(1"')	121.5(2)
C(6')-C(5')-C(4')	120.0(2)
C(6')-C(5')-H(5')	120.0
C(4')-C(5')-H(5')	120.0
N(1')-C(6')-C(5')	121.6(2)
N(1')-C(6')-C(2")	113.9(2)
C(5')-C(6')-C(2")	124.5(2)
N(1")-C(2")-C(3")	122.0(2)
N(1")-C(2")-C(6')	114.8(2)
C(3")-C(2")-C(6')	123.2(2)
C(4")-C(3")-C(2")	118.3(2)
C(4")-C(3")-H(3")	120.9
C(2")-C(3")-H(3")	120.9
C(5")-C(4")-C(3")	119.8(2)
C(5")-C(4")-H(4")	120.1
C(3")-C(4")-H(4")	120.1
C(6")-C(5")-C(4")	118.7(2)
C(6")-C(5")-H(5")	120.7
C(4")-C(5")-H(5")	120.7
N(1")-C(6")-C(5")	122.4(2)
N(1")-C(6")-H(6")	118.8
C(5")-C(6")-H(6")	118.8
C(2"')-C(1"')-C(6"')	116.9(2)
C(2"')-C(1"')-C(4')	121.1(2)
C(6"')-C(1"')-C(4')	121.9(2)
C(3"')-C(2"')-C(1"')	121.7(2)
C(3"')-C(2"')-H(2"')	119.1
C(1"')-C(2"')-H(2"')	119.1
C(4"')-C(3"')-C(2"')	121.2(2)
C(4"')-C(3"')-H(3"')	119.4
C(2"')-C(3"')-H(3"')	119.4
C(3"')-C(4"')-C(5"')	118.0(2)
C(3"')-C(4"')-C(7)	119.2(2)
C(5"')-C(4"')-C(7)	122.7(2)
C(6"')-C(5"')-C(4"')	121.1(3)
C(6"')-C(5"')-H(5"')	119.4
C(4"')-C(5"')-H(5"')	119.4
C(5"')-C(6"')-C(1"')	121.0(2)
C(5"')-C(6"')-H(6"')	119.5
C(1"')-C(6"')-H(6"')	119.5
O-C(7)-C(4"')	110.3(2)
O-C(7)-H(7A)	109.6
C(4"')-C(7)-H(7A)	109.6
O-C(7)-H(7B)	109.6
C(4"')-C(7)-H(7B)	109.6
H(7A)-C(7)-H(7B)	108.1
C(7)-O-C(01)	111.0(2)
O-C(01)-C(02)	107.4(2)
O-C(01)-H(01A)	110.2
C(02)-C(01)-H(01A)	110.2
O-C(01)-H(01B)	110.2

C(02)-C(01)-H(01B)	110.2
H(01A)-C(01)-H(01B)	108.5
N(01")-C(02)-N(01)	108.5(2)
N(01")-C(02)-N(01')	107.8(2)
N(01)-C(02)-N(01')	109.8(2)
N(01")-C(02)-C(01)	110.3(2)
N(01)-C(02)-C(01)	111.5(2)
N(01')-C(02)-C(01)	108.8(2)
C(05)-N(01)-N(02)	111.4(2)
C(05)-N(01)-C(02)	127.9(2)
N(02)-N(01)-C(02)	120.1(2)
C(03)-N(02)-N(01)	104.2(2)
C(05')-N(01')-N(02')	112.4(2)
C(05')-N(01')-C(02)	127.6(2)
N(02')-N(01')-C(02)	119.8(2)
C(03')-N(02')-N(01')	103.8(2)
C(05")-N(01")-N(02")	111.0(3)
C(05")-N(01")-C(02)	129.5(3)
N(02")-N(01")-C(02)	117.7(2)
C(03")-N(02")-N(01")	104.7(3)
N(02)-C(03)-C(04)	112.5(3)
N(02)-C(03)-H(03)	123.8
C(04)-C(03)-H(03)	123.8
C(05)-C(04)-C(03)	104.8(3)
C(05)-C(04)-H(04)	127.6
C(03)-C(04)-H(04)	127.6
N(01)-C(05)-C(04)	107.1(3)
N(01)-C(05)-H(05)	126.4
C(04)-C(05)-H(05)	126.4
N(02')-C(03')-C(04')	111.9(3)
N(02')-C(03')-H(03')	124.1
C(04')-C(03')-H(03')	124.1
C(05')-C(04')-C(03')	105.2(2)
C(05')-C(04')-H(04')	127.4
C(03')-C(04')-H(04')	127.4
N(01')-C(05')-C(04')	106.7(3)
N(01')-C(05')-H(05')	126.6
C(04')-C(05')-H(05')	126.6
N(02")-C(03")-C(04")	111.9(3)
N(02")-C(03")-H(03")	124.0
C(04")-C(03")-H(03")	124.0
C(05")-C(04")-C(03")	105.6(3)
C(05")-C(04")-H(04")	127.2
C(03")-C(04")-H(04")	127.2
C(04")-C(05")-N(01")	106.8(3)
C(04")-C(05")-H(05")	126.6
N(01")-C(05")-H(05")	126.6
N-C-C'	178.8(5)
C-C'-H'1	109.5
C-C'-H'2	109.5
H'1-C'-H'2	109.5
C-C'-H'3	109.5
H'1-C'-H'3	109.5
H'2-C'-H'3	109.5

Symmetry transformations used to generate equivalent atoms:

Table A2.101. Anisotropic displacement parameters ($Å^2$ x 10^3) for [Zn(pzt)Cl_2], **4.8.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Zn	20(1)	15(1)	19(1)	-2(1)	-3(1)	1(1)
N(1)	16(1)	15(1)	23(1)	-1(1)	-1(1)	-2(1)
N(1')	15(1)	13(1)	23(1)	-3(1)	-2(1)	-2(1)
N(1")	20(1)	17(1)	19(1)	-2(1)	-2(1)	0(1)
C(2)	14(1)	15(1)	26(1)	-1(1)	-1(1)	-3(1)
C(3)	20(1)	19(1)	27(1)	-5(1)	0(1)	-3(1)
C(4)	19(1)	16(1)	35(2)	-5(1)	4(1)	-1(1)
C(5)	17(1)	14(1)	36(2)	3(1)	0(1)	0(1)
C(6)	17(1)	18(1)	27(1)	3(1)	-2(1)	-3(1)
C(2')	16(1)	13(1)	23(1)	-3(1)	0(1)	-2(1)
C(3')	20(1)	16(1)	23(1)	-6(1)	-1(1)	-2(1)
C(4')	17(1)	16(1)	22(1)	-3(1)	-2(1)	-4(1)
C(5')	19(1)	16(1)	24(1)	-3(1)	-3(1)	0(1)
C(6')	17(1)	13(1)	23(1)	-3(1)	-3(1)	-1(1)
C(2")	19(1)	16(1)	20(1)	-2(1)	-1(1)	-2(1)
C(3")	23(1)	19(1)	23(1)	-3(1)	-7(1)	0(1)
C(4")	27(1)	19(1)	26(1)	-5(1)	-1(1)	4(1)
C(5")	30(1)	22(1)	22(1)	-7(1)	-1(1)	0(1)
C(6")	26(1)	22(1)	20(1)	-2(1)	-4(1)	-1(1)
C(1"')	20(1)	17(1)	23(1)	-5(1)	-4(1)	-2(1)
C(2"')	38(2)	25(1)	29(2)	-12(1)	-12(1)	11(1)
C(3"')	33(2)	27(1)	33(2)	-8(1)	-15(1)	10(1)
C(4"')	25(1)	24(1)	25(1)	-2(1)	-8(1)	0(1)
C(5"')	43(2)	52(2)	28(2)	-19(1)	-13(1)	20(1)
C(6"')	45(2)	44(2)	32(2)	-16(1)	-17(1)	27(1)
C(7)	32(2)	36(2)	27(2)	-8(1)	-10(1)	8(1)
O	30(1)	57(1)	19(1)	-8(1)	-8(1)	11(1)
C(01)	31(2)	43(2)	19(1)	-4(1)	-8(1)	10(1)
C(02)	30(1)	35(2)	20(1)	-2(1)	-7(1)	9(1)
N(01)	39(1)	41(1)	20(1)	3(1)	-1(1)	13(1)
N(02)	34(1)	43(2)	29(1)	6(1)	1(1)	6(1)
N(01')	27(1)	31(1)	20(1)	-1(1)	-8(1)	3(1)
N(02')	29(1)	35(1)	30(1)	-3(1)	-9(1)	7(1)
N(01")	26(1)	48(2)	23(1)	-5(1)	-7(1)	4(1)
N(02")	41(2)	40(2)	41(2)	-9(1)	-9(1)	-4(1)
C(03)	39(2)	40(2)	39(2)	7(1)	-4(1)	8(1)
C(04)	62(2)	44(2)	37(2)	4(1)	3(2)	24(2)
C(05)	67(2)	51(2)	26(2)	7(1)	11(2)	25(2)
C(03')	37(2)	36(2)	35(2)	-4(1)	-19(1)	6(1)
C(04')	49(2)	39(2)	22(1)	-3(1)	-14(1)	0(1)
C(05')	37(2)	31(2)	23(1)	-2(1)	-5(1)	1(1)
C(03")	43(2)	65(2)	43(2)	-17(2)	-3(2)	-18(2)
C(04")	31(2)	98(3)	48(2)	-14(2)	-8(2)	-14(2)
C(05")	28(2)	83(3)	36(2)	3(2)	-7(1)	13(2)
Cl(1)	20(1)	17(1)	29(1)	-4(1)	-9(1)	1(1)
Cl(2)	40(1)	28(1)	24(1)	5(1)	4(1)	8(1)
N	116(3)	52(2)	114(3)	-36(2)	78(3)	-30(2)
C	63(2)	30(2)	54(2)	-8(1)	24(2)	-12(2)
C'	67(2)	41(2)	44(2)	-6(2)	15(2)	6(2)

Table A2.102. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for [Zn(pzt)Cl_2], **4.8.**

	x	y	z	U(eq)
H(3)	6943	4811	3815	26
H(4)	8709	3225	4210	28
H(5)	9465	3211	5405	27
H(6)	8495	4782	6172	25
H(3')	5283	6347	3552	23
H(5')	2728	9335	4359	23
H(3")	2025	10372	5364	25
H(4")	1637	11471	6447	29
H(5")	3087	10856	7434	29
H(6")	4908	9180	7317	27
H(2"')	1598	9493	3446	36
H(3"')	438	9803	2365	37
H(5"')	3265	7048	1470	49
H(6"')	4440	6723	2555	48
H(7A)	-221	8289	1247	37
H(7B)	657	9509	1019	37
H(01A)	334	8837	-183	37
H(01B)	-82	7493	104	37
H(03)	2143	3888	305	49
H(04)	3977	3693	-887	60
H(05)	4041	5769	-1486	62
H(03')	-1998	7798	-2092	42
H(04')	515	7663	-3015	43
H(05')	2840	7545	-2270	37
H(03")	4739	10644	-1215	59
H(04")	6710	9108	-726	69
H(05")	5276	7217	-467	60
H'1	9719	4666	1665	80
H'2	11207	5045	2052	80
H'3	10629	5841	1360	80

Table A2.103. Hydrogen bonds for [Zn(pzt)Cl_2], **4.8** [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

Table A2.104. Crystal data and structure refinement for $[Ru(tpt)_2](PF_6)_2$, **6.2.**

Identification code	**6.2**
Empirical formula	C36 H24 F12 N12 P2 Ru
Formula weight	1015.68
Temperature	358(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, *P c c a*
Unit cell dimensions	a = 21.474(11) Å alpha = 90°. b = 11.008(6) Å beta = 90°. c = 16.044(8) Å gamma = 90°.
Volume	3793(3) $Å^3$
Z, Calculated density	4, 1.779 Mg/m^3
Absorption coefficient	0.606 mm^{-1}
F(000)	2024
Crystal size	0.61 x 0.29 x 0.08 mm
Theta range for data collection	1.85 to 26.40°.
Limiting indices	-26<=h<=26, -13<=k<=11, -18<=l<=19
Reflections collected / unique	21050 / 3864 [R(int) = 0.0439]
Completeness to theta = 26.40	99.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9531 and 0.7089
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3864 / 0 / 287
Goodness-of-fit on F^2	1.156
Final R indices [I>2sigma(I)]	R1 = 0.0584, wR2 = 0.1295
R indices (all data)	R1 = 0.0797, wR2 = 0.1373
Largest diff. peak and hole	1.536 and -1.085 eA^{-3}

Table A2.105. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $[Ru(tpt)_2](PF_6)_2$, **6.2.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ru	2500	0	963(1)	27(1)
N(1)	3145(2)	1289(3)	975(3)	28(1)
N(2)	3529(2)	3145(4)	454(3)	33(1)
N(3)	4059(2)	2107(4)	1556(3)	33(1)
N(4)	3137(2)	-631(4)	1861(3)	28(1)
N(5)	2825(2)	-1222(4)	47(3)	31(1)
N(6)	5289(3)	5979(5)	1237(4)	60(2)
C(1)	3108(2)	2261(5)	456(3)	30(1)
C(2)	3987(2)	3039(5)	1015(4)	34(1)
C(3)	3633(2)	1240(4)	1497(3)	29(1)
C(4)	3638(2)	128(5)	2005(3)	29(1)
C(5)	4114(2)	-155(5)	2536(3)	34(1)
C(6)	4100(3)	-1253(5)	2958(4)	42(1)
C(7)	3602(3)	-2015(5)	2837(4)	40(1)
C(8)	3129(3)	-1690(5)	2295(4)	36(1)
C(9)	3338(3)	-1138(5)	-413(4)	38(1)
C(10)	3504(3)	-2032(5)	-973(4)	45(2)
C(11)	3133(3)	-3047(5)	-1077(4)	45(2)
C(12)	2599(3)	-3136(5)	-615(3)	36(1)
C(13)	2445(3)	-2224(4)	-70(3)	32(1)
C(14)	4443(3)	4046(5)	1071(4)	39(1)
C(15)	4316(3)	5181(5)	720(4)	49(2)
C(16)	4748(3)	6107(6)	821(5)	56(2)
C(17)	5399(3)	4873(7)	1545(5)	60(2)
C(18)	4995(3)	3901(5)	1482(4)	46(2)
P(1)	2500	5000	2020(1)	29(1)
F(11)	2934(1)	3804(3)	2031(2)	37(1)
F(12)	2928(2)	5580(3)	2741(2)	40(1)
F(13)	2926(2)	5604(3)	1330(2)	50(1)
P(2)	5000	10000	0	31(1)
F(21)	5229(1)	10763(3)	790(2)	41(1)
F(22)	4579(2)	9182(3)	601(2)	44(1)
F(23)	5573(1)	9068(3)	125(2)	37(1)

Table A2.106. Selected bond lengths [Å] and angles [°] for $[Ru(tpt)_2](PF_6)_2$, **6.2.**

Ru–N(1)#1	1.983(4)
Ru–N(1)	1.983(4)
Ru–N(4)#1	2.104(4)
Ru–N(4)	2.104(4)
Ru–N(5)#1	2.111(4)
Ru–N(5)	2.111(4)
N(1)#1–Ru–N(1)	178.9(3)
N(1)#1–Ru–N(4)#1	77.00(17)
N(1)–Ru–N(4)#1	102.22(16)
N(1)#1–Ru–N(4)	102.22(16)
N(1)–Ru–N(4)	77.00(17)
N(4)#1–Ru–N(4)	93.6(2)
N(1)#1–Ru–N(5)#1	103.38(17)
N(1)–Ru–N(5)#1	77.41(18)
N(4)#1–Ru–N(5)#1	92.97(17)
N(4)–Ru–N(5)#1	154.38(16)
N(1)#1–Ru–N(5)	77.41(18)
N(1)–Ru–N(5)	103.38(17)
N(4)#1–Ru–N(5)	154.38(16)
N(4)–Ru–N(5)	92.97(17)
N(5)#1–Ru–N(5)	91.7(2)

Symmetry transformations used to generate equivalent atoms:

Table A2.107. Bond lengths [Å] and angles [°] for $[Ru(tpt)_2](PF_6)_2$, **6.2.**

Ru-N(1)#1	1.983(4)
Ru-N(1)	1.983(4)
Ru-N(4)#1	2.104(4)
Ru-N(4)	2.104(4)
Ru-N(5)#1	2.111(4)
Ru-N(5)	2.111(4)
N(1)-C(3)	1.342(7)
N(1)-C(1)	1.359(7)
N(2)-C(1)	1.329(6)
N(2)-C(2)	1.338(7)
N(3)-C(3)	1.326(6)
N(3)-C(2)	1.353(7)
N(4)-C(8)	1.358(7)
N(4)-C(4)	1.381(6)
N(5)-C(9)	1.328(7)
N(5)-C(13)	1.385(6)
N(6)-C(17)	1.335(9)
N(6)-C(16)	1.348(9)
C(1)-C(13)#1	1.458(8)
C(2)-C(14)	1.483(7)
C(3)-C(4)	1.470(7)
C(4)-C(5)	1.367(7)
C(5)-C(6)	1.386(8)
C(5)-H(5)	0.9300
C(6)-C(7)	1.372(8)
C(6)-H(6)	0.9300
C(7)-C(8)	1.385(7)
C(7)-H(7)	0.9300
C(8)-H(8)	0.9300
C(9)-C(10)	1.379(8)
C(9)-H(9)	0.9300
C(10)-C(11)	1.382(8)
C(10)-H(10)	0.9300
C(11)-C(12)	1.369(8)
C(11)-H(11)	0.9300
C(12)-C(13)	1.372(7)
C(12)-H(12)	0.9300
C(13)-C(1)#1	1.458(8)
C(14)-C(18)	1.365(9)
C(14)-C(15)	1.397(8)
C(15)-C(16)	1.388(8)
C(15)-H(15)	0.9300
C(16)-H(16)	0.9300
C(17)-C(18)	1.382(8)
C(17)-H(17)	0.9300
C(18)-H(18)	0.9300
P(1)-F(13)#2	1.582(3)
P(1)-F(13)	1.582(3)
P(1)-F(12)	1.610(3)
P(1)-F(12)#2	1.610(3)
P(1)-F(11)#2	1.613(3)
P(1)-F(11)	1.613(3)
P(2)-F(21)#3	1.599(3)
P(2)-F(21)	1.599(3)
P(2)-F(22)	1.599(3)
P(2)-F(22)#3	1.599(3)

P(2)-F(23)#3	1.614(3)
P(2)-F(23)	1.614(3)
N(1)#1-Ru-N(1)	178.9(3)
N(1)#1-Ru-N(4)#1	77.00(17)
N(1)-Ru-N(4)#1	102.22(16)
N(1)#1-Ru-N(4)	102.22(16)
N(1)-Ru-N(4)	77.00(17)
N(4)#1-Ru-N(4)	93.6(2)
N(1)#1-Ru-N(5)#1	103.38(17)
N(1)-Ru-N(5)#1	77.41(18)
N(4)#1-Ru-N(5)#1	92.97(17)
N(4)-Ru-N(5)#1	154.38(16)
N(1)#1-Ru-N(5)	77.41(18)
N(1)-Ru-N(5)	103.38(17)
N(4)#1-Ru-N(5)	154.38(16)
N(4)-Ru-N(5)	92.97(17)
N(5)#1-Ru-N(5)	91.7(2)
C(3)-N(1)-C(1)	117.4(4)
C(3)-N(1)-Ru	121.5(3)
C(1)-N(1)-Ru	121.1(4)
C(1)-N(2)-C(2)	115.7(5)
C(3)-N(3)-C(2)	114.9(5)
C(8)-N(4)-C(4)	116.3(5)
C(8)-N(4)-Ru	128.8(3)
C(4)-N(4)-Ru	114.9(3)
C(9)-N(5)-C(13)	117.9(5)
C(9)-N(5)-Ru	128.1(4)
C(13)-N(5)-Ru	114.0(4)
C(17)-N(6)-C(16)	115.5(6)
N(2)-C(1)-N(1)	122.6(5)
N(2)-C(1)-C(13)#1	125.0(5)
N(1)-C(1)-C(13)#1	112.4(4)
N(2)-C(2)-N(3)	125.6(5)
N(2)-C(2)-C(14)	117.4(5)
N(3)-C(2)-C(14)	116.9(5)
N(3)-C(3)-N(1)	123.7(5)
N(3)-C(3)-C(4)	123.7(5)
N(1)-C(3)-C(4)	112.6(4)
C(5)-C(4)-N(4)	123.3(5)
C(5)-C(4)-C(3)	122.7(5)
N(4)-C(4)-C(3)	113.9(4)
C(4)-C(5)-C(6)	119.2(5)
C(4)-C(5)-H(5)	120.4
C(6)-C(5)-H(5)	120.4
C(7)-C(6)-C(5)	118.7(5)
C(7)-C(6)-H(6)	120.7
C(5)-C(6)-H(6)	120.7
C(6)-C(7)-C(8)	120.2(5)
C(6)-C(7)-H(7)	119.9
C(8)-C(7)-H(7)	119.9
N(4)-C(8)-C(7)	122.3(5)
N(4)-C(8)-H(8)	118.8
C(7)-C(8)-H(8)	118.8
N(5)-C(9)-C(10)	121.8(5)
N(5)-C(9)-H(9)	119.1
C(10)-C(9)-H(9)	119.1
C(9)-C(10)-C(11)	120.4(6)
C(9)-C(10)-H(10)	119.8
C(11)-C(10)-H(10)	119.8
C(12)-C(11)-C(10)	118.4(6)

C(12)-C(11)-H(11)	120.8
C(10)-C(11)-H(11)	120.8
C(13)-C(12)-C(11)	119.6(5)
C(13)-C(12)-H(12)	120.2
C(11)-C(12)-H(12)	120.2
C(12)-C(13)-N(5)	121.9(5)
C(12)-C(13)-C(1)#1	123.0(5)
N(5)-C(13)-C(1)#1	115.1(5)
C(18)-C(14)-C(15)	118.0(5)
C(18)-C(14)-C(2)	121.0(5)
C(15)-C(14)-C(2)	121.0(6)
C(16)-C(15)-C(14)	118.6(6)
C(16)-C(15)-H(15)	120.7
C(14)-C(15)-H(15)	120.7
N(6)-C(16)-C(15)	123.9(7)
N(6)-C(16)-H(16)	118.0
C(15)-C(16)-H(16)	118.0
N(6)-C(17)-C(18)	124.6(7)
N(6)-C(17)-H(17)	117.7
C(18)-C(17)-H(17)	117.7
C(14)-C(18)-C(17)	119.3(6)
C(14)-C(18)-H(18)	120.4
C(17)-C(18)-H(18)	120.4
F(13)#2-P(1)-F(13)	91.3(3)
F(13)#2-P(1)-F(12)	178.2(2)
F(13)-P(1)-F(12)	90.37(19)
F(13)#2-P(1)-F(12)#2	90.37(19)
F(13)-P(1)-F(12)#2	178.2(2)
F(12)-P(1)-F(12)#2	88.0(3)
F(13)#2-P(1)-F(11)#2	90.98(17)
F(13)-P(1)-F(11)#2	89.91(18)
F(12)-P(1)-F(11)#2	89.87(16)
F(12)#2-P(1)-F(11)#2	89.21(16)
F(13)#2-P(1)-F(11)	89.91(18)
F(13)-P(1)-F(11)	90.98(17)
F(12)-P(1)-F(11)	89.21(16)
F(12)#2-P(1)-F(11)	89.87(16)
F(11)#2-P(1)-F(11)	178.7(3)
F(21)#3-P(2)-F(21)	180.00(14)
F(21)#3-P(2)-F(22)	90.46(18)
F(21)-P(2)-F(22)	89.54(18)
F(21)#3-P(2)-F(22)#3	89.54(18)
F(21)-P(2)-F(22)#3	90.46(18)
F(22)-P(2)-F(22)#3	180.000(1)
F(21)#3-P(2)-F(23)#3	90.07(16)
F(21)-P(2)-F(23)#3	89.93(16)
F(22)-P(2)-F(23)#3	90.11(16)
F(22)#3-P(2)-F(23)#3	89.89(16)
F(21)#3-P(2)-F(23)	89.93(16)
F(21)-P(2)-F(23)	90.07(16)
F(22)-P(2)-F(23)	89.89(16)
F(22)#3-P(2)-F(23)	90.11(16)
F(23)#3-P(2)-F(23)	180.0(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1/2,-y,z #2 -x+1/2,-y+1,z #3 -x+1,-y+2,-z

Table A2.108. Anisotropic displacement parameters (A^2 x 10^3) for $[Ru(tpt)_2](PF_6)_2$, **6.2.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Ru	30(1)	18(1)	34(1)	0	0	-5(1)
N(1)	33(2)	18(2)	32(2)	-2(2)	8(2)	-2(2)
N(2)	38(2)	20(2)	40(3)	-5(2)	20(2)	-5(2)
N(3)	31(2)	26(2)	41(3)	-11(2)	8(2)	-5(2)
N(4)	27(2)	24(2)	34(2)	-3(2)	-1(2)	-4(2)
N(5)	37(2)	22(2)	35(2)	-1(2)	-6(2)	-3(2)
N(6)	61(4)	41(3)	79(4)	-14(3)	21(3)	-15(3)
C(1)	35(3)	23(2)	31(3)	-4(2)	12(2)	-4(2)
C(2)	35(3)	27(3)	42(3)	-8(2)	17(3)	-4(2)
C(3)	28(3)	26(2)	32(3)	-8(2)	8(2)	-2(2)
C(4)	32(3)	23(3)	32(3)	-10(2)	7(2)	-6(2)
C(5)	28(2)	36(3)	38(3)	-13(3)	1(2)	-5(2)
C(6)	37(3)	43(3)	45(4)	-7(3)	-11(3)	3(3)
C(7)	45(3)	35(3)	41(3)	3(3)	-14(3)	-3(3)
C(8)	38(3)	26(3)	45(4)	4(2)	-5(3)	-7(2)
C(9)	39(3)	28(3)	45(3)	-2(3)	-4(3)	-7(2)
C(10)	45(3)	43(3)	48(4)	-14(3)	0(3)	-3(3)
C(11)	55(4)	33(3)	47(4)	-15(3)	-7(3)	3(3)
C(12)	39(3)	29(3)	41(3)	-2(2)	-13(3)	-3(2)
C(13)	38(3)	23(2)	35(3)	3(2)	-13(3)	-1(2)
C(14)	41(3)	27(3)	49(4)	-11(3)	19(3)	-9(2)
C(15)	47(3)	31(3)	69(4)	-9(3)	15(3)	-6(3)
C(16)	60(4)	28(3)	80(5)	-6(3)	18(4)	-10(3)
C(17)	58(4)	50(4)	73(5)	-10(4)	12(4)	-16(4)
C(18)	47(3)	35(3)	56(4)	-7(3)	6(3)	-14(3)
P(1)	37(1)	23(1)	29(1)	0	0	4(1)
F(11)	44(2)	26(2)	40(2)	-4(1)	7(1)	7(1)
F(12)	44(2)	27(2)	47(2)	-3(2)	-10(2)	5(1)
F(13)	62(2)	40(2)	48(2)	11(2)	18(2)	5(2)
P(2)	34(1)	28(1)	32(1)	-4(1)	2(1)	5(1)
F(21)	40(2)	40(2)	43(2)	-14(2)	-5(2)	8(1)
F(22)	44(2)	37(2)	51(2)	4(2)	9(2)	3(2)
F(23)	38(2)	35(2)	37(2)	-5(1)	0(1)	10(1)

Table A2.109. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for $[Ru(tpt)_2](PF_6)_2$, **6.2.**

	x	y	z	U(eq)
H(5)	4443	382	2613	41
H(6)	4422	-1470	3316	50
H(7)	3583	-2752	3119	48
H(8)	2794	-2216	2225	43
H(9)	3591	-459	-356	45
H(10)	3868	-1951	-1282	55
H(11)	3244	-3655	-1451	54
H(12)	2341	-3810	-672	44
H(15)	3949	5313	425	59
H(16)	4658	6861	589	67
H(17)	5774	4748	1822	72
H(18)	5097	3155	1717	55

Table A2.110. Hydrogen bonds for $[Ru(tpt)_2](PF_6)_2$, **6.2** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

Table A2.111. Crystal data and structure refinement for $[Fe(tpt)Cl_2]_2(\mu\text{-}O).2(H_2O)$, **6.4**

Identification code	9rzgr(sad)
Empirical formula	C36 H26 Cl4 Fe2 N12 O3
Formula weight	928.19
Temperature	84(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, *P*2(1)/*c*
Unit cell dimensions	a = 10.8726(11) Å alpha = 90°. b = 15.8656(16) Å beta = 93.135(2)°. c = 22.747(2) Å gamma = 90°.
Volume	3918.0(7) $Å^3$
Z, Calculated density	4, 1.574 Mg/m^3
Absorption coefficient	1.067 mm^{-1}
F(000)	1880
Crystal size	0.45 x 0.35 x 0.10 mm
Theta range for data collection	2.27 to 26.43°.
Limiting indices	-13<=h<=13, -19<=k<=18, -28<=l<=28
Reflections collected / unique	34106 / 7995 [R(int) = 0.0411]
Completeness to theta = 26.43	99.3 %
Absorption correction	None
Max. and min. transmission	0.9008 and 0.6452
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7995 / 0 / 525
Goodness-of-fit on F^2	1.022
Final R indices [I>2sigma(I)]	R1 = 0.0419, wR2 = 0.1143
R indices (all data)	R1 = 0.0608, wR2 = 0.1238
Largest diff. peak and hole	1.475 and -0.451 $eÅ^{-3}$

Table A2.112. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $[Fe(tpt)Cl_2]_2(\mu\text{-}O).2(H_2O)$, **6.4.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Fe	3467(1)	3017(1)	7735(1)	19(1)
N(1)	2569(2)	3140(2)	8536(1)	21(1)
N(2)	1008(2)	3761(2)	9072(1)	22(1)
N(3)	2227(2)	2621(2)	9480(1)	24(1)
N(4)	4318(2)	2093(2)	8355(1)	21(1)
N(5)	2068(2)	3998(2)	7600(1)	22(1)
N(6)	-181(3)	3852(2)	10113(1)	40(1)
C(1)	1651(3)	3692(2)	8594(1)	20(1)
C(2)	1326(3)	3206(2)	9499(1)	23(1)
C(3)	2825(3)	2619(2)	8990(1)	21(1)
C(4)	3846(3)	2025(2)	8893(1)	21(1)
C(5)	4262(3)	1449(2)	9311(1)	26(1)
C(6)	5217(3)	913(2)	9176(1)	29(1)
C(7)	5709(3)	980(2)	8629(1)	29(1)
C(8)	5243(3)	1577(2)	8226(1)	26(1)
C(9)	1390(3)	4212(2)	8061(1)	21(1)
C(10)	533(3)	4854(2)	8028(1)	28(1)
C(11)	369(3)	5298(2)	7505(2)	33(1)
C(12)	1041(3)	5072(2)	7029(2)	34(1)
C(13)	1879(3)	4418(2)	7091(1)	27(1)
C(14)	629(3)	3222(2)	10048(1)	26(1)
C(15)	870(3)	2609(2)	10457(1)	30(1)
C(16)	262(4)	2631(3)	10966(2)	40(1)
C(17)	-576(3)	3263(3)	11054(2)	37(1)
C(18)	-778(3)	3866(3)	10626(2)	40(1)
Cl(1)	1846(1)	2013(1)	7488(1)	24(1)
Cl(2)	4812(1)	4115(1)	8108(1)	34(1)
Fe'	5039(1)	2809(1)	6423(1)	25(1)
N(1')	5982(3)	2654(2)	5635(1)	26(1)
N(2')	6268(3)	1903(2)	4762(1)	29(1)
N(3')	7535(3)	3101(2)	5021(1)	30(1)
N(4')	6395(2)	3826(2)	6400(1)	27(1)
N(5')	4217(2)	1717(2)	5960(1)	25(1)
N(6')	8774(3)	2873(2)	4024(1)	38(1)
C(1')	5687(3)	2039(2)	5256(1)	26(1)
C(2')	7187(3)	2458(2)	4665(1)	29(1)
C(3')	6889(3)	3173(2)	5501(1)	27(1)
C(4')	7116(3)	3857(2)	5934(1)	28(1)
C(5')	7965(3)	4492(2)	5862(2)	34(1)
C(6')	8068(3)	5124(2)	6282(2)	38(1)
C(7')	7337(3)	5091(2)	6763(2)	34(1)
C(8')	6513(3)	4432(2)	6805(2)	29(1)
C(9')	4655(3)	1510(2)	5429(1)	26(1)
C(10')	4159(3)	861(2)	5085(1)	28(1)
C(11')	3217(3)	386(2)	5299(2)	34(1)
C(12')	2793(3)	582(2)	5846(2)	35(1)
C(13')	3311(3)	1257(2)	6160(2)	29(1)
C(14')	7826(3)	2351(2)	4110(1)	32(1)
C(15')	7409(4)	1765(3)	3695(2)	43(1)
C(16')	7969(4)	1726(3)	3164(2)	50(1)
C(17')	8933(4)	2250(3)	3070(2)	44(1)
C(18')	9319(4)	2808(3)	3510(2)	42(1)

Cl(1')	6668(1)	1957(1)	6867(1)	32(1)
Cl(2')	3707(1)	3754(1)	5850(1)	32(1)
O	4214(2)	2889(1)	7070(1)	27(1)
O(1)	3336(2)	5296(2)	8930(1)	33(1)
O(2')	1078(8)	4519(10)	5617(4)	76(6)
O(2)	9818(8)	4298(5)	4538(3)	162(5)

Table A2.113. Selected bond lengths [Å] and angles [°] for $[Fe(tpt)Cl_2]_2(\mu\text{-}O).2(H_2O)$, **6.4**.

Fe-O	1.767(2)
Fe-N(1)	2.124(2)
Fe-N(5)	2.186(3)
Fe-N(4)	2.202(3)
Fe-Cl(2)	2.3999(9)
Fe-Cl(1)	2.4187(9)
Fe'-O	1.768(2)
Fe'-N(1')	2.127(3)
Fe'-N(4')	2.189(3)
Fe'-N(5')	2.193(3)
Fe'-Cl(1')	2.4051(10)
Fe'-Cl(2')	2.4162(10)
O-Fe-N(1)	178.67(10)
O-Fe-N(5)	108.07(10)
N(1)-Fe-N(5)	72.92(10)
O-Fe-N(4)	105.96(10)
N(1)-Fe-N(4)	73.03(10)
N(5)-Fe-N(4)	145.95(10)
O-Fe-Cl(2)	94.97(8)
N(1)-Fe-Cl(2)	85.96(7)
N(5)-Fe-Cl(2)	86.49(7)
N(4)-Fe-Cl(2)	91.71(7)
O-Fe-Cl(1)	95.12(8)
N(1)-Fe-Cl(1)	84.01(7)
N(5)-Fe-Cl(1)	86.78(7)
N(4)-Fe-Cl(1)	89.16(7)
Cl(2)-Fe-Cl(1)	169.21(3)

Symmetry transformations used to generate equivalent atoms:

Table A2.114. Bond lengths [Å] and angles [°] for $[Fe(tpt)Cl_2]_2(\mu\text{-}O).2(H_2O)$, **6.4.**

Fe-O	1.767(2)
Fe-N(1)	2.124(2)
Fe-N(5)	2.186(3)
Fe-N(4)	2.202(3)
Fe-Cl(2)	2.3999(9)
Fe-Cl(1)	2.4187(9)
N(1)-C(3)	1.339(4)
N(1)-C(1)	1.340(4)
N(2)-C(1)	1.330(4)
N(2)-C(2)	1.342(4)
N(3)-C(3)	1.321(4)
N(3)-C(2)	1.351(4)
N(4)-C(8)	1.341(4)
N(4)-C(4)	1.357(4)
N(5)-C(13)	1.342(4)
N(5)-C(9)	1.357(4)
N(6)-C(14)	1.346(5)
N(6)-C(18)	1.366(4)
C(1)-C(9)	1.481(4)
C(2)-C(14)	1.496(4)
C(3)-C(4)	1.481(4)
C(4)-C(5)	1.378(4)
C(5)-C(6)	1.389(5)
C(5)-H(5)	0.9500
C(6)-C(7)	1.385(5)
C(6)-H(6)	0.9500
C(7)-C(8)	1.396(5)
C(7)-H(7)	0.9500
C(8)-H(8)	0.9500
C(9)-C(10)	1.379(4)
C(10)-C(11)	1.387(5)
C(10)-H(10)	0.9500
C(11)-C(12)	1.386(5)
C(11)-H(11)	0.9500
C(12)-C(13)	1.383(5)
C(12)-H(12)	0.9500
C(13)-H(13)	0.9500
C(14)-C(15)	1.362(5)
C(15)-C(16)	1.365(5)
C(15)-H(15)	0.9500
C(16)-C(17)	1.377(6)
C(16)-H(16)	0.9500
C(17)-C(18)	1.373(5)
C(17)-H(17)	0.9500
C(18)-H(18)	0.9500
Fe'-O	1.768(2)
Fe'-N(1')	2.127(3)
Fe'-N(4')	2.189(3)
Fe'-N(5')	2.193(3)
Fe'-Cl(1')	2.4051(10)
Fe'-Cl(2')	2.4162(10)
N(1')-C(1')	1.330(4)
N(1')-C(3')	1.333(4)
N(2')-C(1')	1.336(4)
N(2')-C(2')	1.358(5)
N(3')-C(3')	1.335(4)
N(3')-C(2')	1.344(5)

N(4')-C(8')	1.332(4)
N(4')-C(4')	1.355(4)
N(5')-C(13')	1.327(4)
N(5')-C(9')	1.362(4)
N(6')-C(18')	1.344(4)
N(6')-C(14')	1.345(5)
C(1')-C(9')	1.472(5)
C(2')-C(14')	1.484(4)
C(3')-C(4')	1.477(5)
C(4')-C(5')	1.382(5)
C(5')-C(6')	1.385(5)
C(5')-H(5')	0.9500
C(6')-C(7')	1.388(5)
C(6')-H(6')	0.9500
C(7')-C(8')	1.384(5)
C(7')-H(7')	0.9500
C(8')-H(8')	0.9500
C(9')-C(10')	1.384(4)
C(10')-C(11')	1.382(5)
C(10')-H(10')	0.9500
C(11')-C(12')	1.385(5)
C(11')-H(11')	0.9500
C(12')-C(13')	1.389(5)
C(12')-H(12')	0.9500
C(13')-H(13')	0.9500
C(14')-C(15')	1.383(5)
C(15')-C(16')	1.385(5)
C(15')-H(15')	0.9500
C(16')-C(17')	1.364(6)
C(16')-H(16')	0.9500
C(17')-C(18')	1.384(6)
C(17')-H(17')	0.9500
C(18')-H(18')	0.9500
O(1)-H(1A)	1.0522
O(1)-H(1B)	1.0503

O-Fe-N(1)	178.67(10)
O-Fe-N(5)	108.07(10)
N(1)-Fe-N(5)	72.92(10)
O-Fe-N(4)	105.96(10)
N(1)-Fe-N(4)	73.03(10)
N(5)-Fe-N(4)	145.95(10)
O-Fe-Cl(2)	94.97(8)
N(1)-Fe-Cl(2)	85.96(7)
N(5)-Fe-Cl(2)	86.49(7)
N(4)-Fe-Cl(2)	91.71(7)
O-Fe-Cl(1)	95.12(8)
N(1)-Fe-Cl(1)	84.01(7)
N(5)-Fe-Cl(1)	86.78(7)
N(4)-Fe-Cl(1)	89.16(7)
Cl(2)-Fe-Cl(1)	169.21(3)
C(3)-N(1)-C(1)	117.0(3)
C(3)-N(1)-Fe	121.2(2)
C(1)-N(1)-Fe	121.6(2)
C(1)-N(2)-C(2)	114.4(3)
C(3)-N(3)-C(2)	114.9(3)
C(8)-N(4)-C(4)	118.3(3)
C(8)-N(4)-Fe	124.0(2)
C(4)-N(4)-Fe	117.6(2)
C(13)-N(5)-C(9)	118.5(3)

C(13)-N(5)-Fe	123.2(2)
C(9)-N(5)-Fe	118.2(2)
C(14)-N(6)-C(18)	116.7(3)
N(2)-C(1)-N(1)	124.1(3)
N(2)-C(1)-C(9)	122.6(3)
N(1)-C(1)-C(9)	113.3(3)
N(2)-C(2)-N(3)	125.8(3)
N(2)-C(2)-C(14)	118.1(3)
N(3)-C(2)-C(14)	116.1(3)
N(3)-C(3)-N(1)	123.8(3)
N(3)-C(3)-C(4)	122.4(3)
N(1)-C(3)-C(4)	113.8(3)
N(4)-C(4)-C(5)	123.3(3)
N(4)-C(4)-C(3)	114.1(3)
C(5)-C(4)-C(3)	122.6(3)
C(4)-C(5)-C(6)	118.4(3)
C(4)-C(5)-H(5)	120.8
C(6)-C(5)-H(5)	120.8
C(7)-C(6)-C(5)	118.8(3)
C(7)-C(6)-H(6)	120.6
C(5)-C(6)-H(6)	120.6
C(6)-C(7)-C(8)	119.9(3)
C(6)-C(7)-H(7)	120.0
C(8)-C(7)-H(7)	120.0
N(4)-C(8)-C(7)	121.3(3)
N(4)-C(8)-H(8)	119.3
C(7)-C(8)-H(8)	119.3
N(5)-C(9)-C(10)	122.5(3)
N(5)-C(9)-C(1)	113.9(3)
C(10)-C(9)-C(1)	123.6(3)
C(9)-C(10)-C(11)	118.4(3)
C(9)-C(10)-H(10)	120.8
C(11)-C(10)-H(10)	120.8
C(12)-C(11)-C(10)	119.4(3)
C(12)-C(11)-H(11)	120.3
C(10)-C(11)-H(11)	120.3
C(13)-C(12)-C(11)	119.1(3)
C(13)-C(12)-H(12)	120.4
C(11)-C(12)-H(12)	120.4
N(5)-C(13)-C(12)	122.0(3)
N(5)-C(13)-H(13)	119.0
C(12)-C(13)-H(13)	119.0
N(6)-C(14)-C(15)	123.9(3)
N(6)-C(14)-C(2)	118.0(3)
C(15)-C(14)-C(2)	118.1(3)
C(14)-C(15)-C(16)	118.5(3)
C(14)-C(15)-H(15)	120.8
C(16)-C(15)-H(15)	120.8
C(15)-C(16)-C(17)	119.8(4)
C(15)-C(16)-H(16)	120.1
C(17)-C(16)-H(16)	120.1
C(18)-C(17)-C(16)	119.1(3)
C(18)-C(17)-H(17)	120.5
C(16)-C(17)-H(17)	120.5
N(6)-C(18)-C(17)	122.0(4)
N(6)-C(18)-H(18)	119.0
C(17)-C(18)-H(18)	119.0
O-Fe'-N(1')	177.05(11)
O-Fe'-N(4')	109.86(10)
N(1')-Fe'-N(4')	73.07(10)
O-Fe'-N(5')	104.16(10)

N(1')-Fe'-N(5')	72.91(10)
N(4')-Fe'-N(5')	145.95(10)
O-Fe'-Cl(1')	95.00(8)
N(1')-Fe'-Cl(1')	84.87(8)
N(4')-Fe'-Cl(1')	86.72(8)
N(5')-Fe'-Cl(1')	91.64(8)
O-Fe'-Cl(2')	95.03(8)
N(1')-Fe'-Cl(2')	85.37(8)
N(4')-Fe'-Cl(2')	85.08(8)
N(5')-Fe'-Cl(2')	90.85(8)
Cl(1')-Fe'-Cl(2')	168.74(4)
C(1')-N(1')-C(3')	117.4(3)
C(1')-N(1')-Fe'	121.4(2)
C(3')-N(1')-Fe'	121.2(2)
C(1')-N(2')-C(2')	114.7(3)
C(3')-N(3')-C(2')	114.4(3)
C(8')-N(4')-C(4')	118.5(3)
C(8')-N(4')-Fe'	123.8(2)
C(4')-N(4')-Fe'	117.7(2)
C(13')-N(5')-C(9')	118.3(3)
C(13')-N(5')-Fe'	124.1(2)
C(9')-N(5')-Fe'	117.6(2)
C(18')-N(6')-C(14')	117.2(3)
N(1')-C(1')-N(2')	123.8(3)
N(1')-C(1')-C(9')	113.9(3)
N(2')-C(1')-C(9')	122.3(3)
N(3')-C(2')-N(2')	125.4(3)
N(3')-C(2')-C(14')	118.1(3)
N(2')-C(2')-C(14')	116.5(3)
N(1')-C(3')-N(3')	124.3(3)
N(1')-C(3')-C(4')	113.6(3)
N(3')-C(3')-C(4')	122.1(3)
N(4')-C(4')-C(5')	122.5(3)
N(4')-C(4')-C(3')	114.4(3)
C(5')-C(4')-C(3')	123.1(3)
C(4')-C(5')-C(6')	118.4(3)
C(4')-C(5')-H(5')	120.8
C(6')-C(5')-H(5')	120.8
C(5')-C(6')-C(7')	119.3(3)
C(5')-C(6')-H(6')	120.4
C(7')-C(6')-H(6')	120.4
C(8')-C(7')-C(6')	118.9(3)
C(8')-C(7')-H(7')	120.6
C(6')-C(7')-H(7')	120.6
N(4')-C(8')-C(7')	122.4(3)
N(4')-C(8')-H(8')	118.8
C(7')-C(8')-H(8')	118.8
N(5')-C(9')-C(10')	122.4(3)
N(5')-C(9')-C(1')	114.1(3)
C(10')-C(9')-C(1')	123.5(3)
C(11')-C(10')-C(9')	118.7(3)
C(11')-C(10')-H(10')	120.7
C(9')-C(10')-H(10')	120.7
C(10')-C(11')-C(12')	118.9(3)
C(10')-C(11')-H(11')	120.6
C(12')-C(11')-H(11')	120.6
C(11')-C(12')-C(13')	119.3(3)
C(11')-C(12')-H(12')	120.3
C(13')-C(12')-H(12')	120.3
N(5')-C(13')-C(12')	122.4(3)
N(5')-C(13')-H(13')	118.8

C(12')-C(13')-H(13')	118.8
N(6')-C(14')-C(15')	122.7(3)
N(6')-C(14')-C(2')	116.8(3)
C(15')-C(14')-C(2')	120.4(3)
C(16')-C(15')-C(14')	118.8(4)
C(16')-C(15')-H(15')	120.6
C(14')-C(15')-H(15')	120.6
C(17')-C(16')-C(15')	119.2(4)
C(17')-C(16')-H(16')	120.4
C(15')-C(16')-H(16')	120.4
C(16')-C(17')-C(18')	118.8(3)
C(16')-C(17')-H(17')	120.6
C(18')-C(17')-H(17')	120.6
N(6')-C(18')-C(17')	123.2(4)
N(6')-C(18')-H(18')	118.4
C(17')-C(18')-H(18')	118.4
Fe-O-Fe'	176.12(15)
H(1A)-O(1)-H(1B)	106.2

Symmetry transformations used to generate equivalent atoms:

Table A2.115. Anisotropic displacement parameters (A^2 x 10^3) for $[Fe(tpt)Cl_2]_2(\mu\text{-}O).2(H_2O)$, **6.4.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Fe	20(1)	19(1)	21(1)	0(1)	6(1)	2(1)
N(1)	20(1)	22(1)	22(1)	-3(1)	4(1)	1(1)
N(2)	22(1)	25(1)	21(1)	-5(1)	4(1)	0(1)
N(3)	25(1)	27(1)	21(1)	-4(1)	3(1)	0(1)
N(4)	21(1)	20(1)	23(1)	-3(1)	2(1)	2(1)
N(5)	21(1)	19(1)	27(1)	-1(1)	5(1)	2(1)
N(6)	35(2)	55(2)	29(2)	-3(1)	4(1)	8(2)
C(1)	17(2)	19(2)	25(2)	-6(1)	2(1)	0(1)
C(2)	20(2)	26(2)	24(2)	-8(1)	4(1)	-2(1)
C(3)	21(2)	20(2)	22(2)	-4(1)	2(1)	-2(1)
C(4)	22(2)	21(2)	21(1)	-4(1)	1(1)	-1(1)
C(5)	30(2)	27(2)	20(2)	-2(1)	0(1)	0(1)
C(6)	35(2)	25(2)	27(2)	-1(1)	-4(1)	5(1)
C(7)	26(2)	27(2)	34(2)	-3(1)	2(1)	8(1)
C(8)	24(2)	27(2)	26(2)	-4(1)	7(1)	2(1)
C(9)	19(2)	20(2)	24(2)	-3(1)	3(1)	-2(1)
C(10)	23(2)	30(2)	30(2)	-4(1)	6(1)	5(1)
C(11)	30(2)	31(2)	39(2)	2(2)	2(2)	13(2)
C(12)	40(2)	32(2)	31(2)	8(2)	5(2)	8(2)
C(13)	28(2)	26(2)	28(2)	3(1)	8(1)	3(1)
C(14)	23(2)	35(2)	22(2)	-8(1)	3(1)	1(1)
C(15)	34(2)	30(2)	27(2)	-4(1)	13(1)	3(2)
C(16)	42(2)	46(2)	33(2)	1(2)	11(2)	-1(2)
C(17)	31(2)	54(2)	28(2)	-10(2)	12(1)	-2(2)
C(18)	28(2)	60(3)	33(2)	-3(2)	6(1)	11(2)
Cl(1)	24(1)	21(1)	27(1)	1(1)	4(1)	-3(1)
Cl(2)	27(1)	33(1)	42(1)	-12(1)	7(1)	-5(1)
Fe'	28(1)	25(1)	22(1)	-6(1)	10(1)	-6(1)
N(1')	28(2)	28(1)	23(1)	-4(1)	7(1)	0(1)
N(2')	30(2)	35(2)	21(1)	-3(1)	3(1)	8(1)
N(3')	28(2)	40(2)	22(1)	4(1)	7(1)	4(1)
N(4')	26(2)	27(2)	28(1)	-2(1)	7(1)	-2(1)
N(5')	25(2)	25(1)	25(1)	-6(1)	4(1)	1(1)
N(6')	41(2)	42(2)	33(2)	9(1)	15(1)	12(2)
C(1')	25(2)	29(2)	23(2)	-3(1)	2(1)	7(1)
C(2')	26(2)	39(2)	23(2)	4(1)	4(1)	11(2)
C(3')	23(2)	33(2)	27(2)	3(1)	4(1)	1(1)
C(4')	28(2)	32(2)	26(2)	2(1)	4(1)	-1(2)
C(5')	29(2)	38(2)	37(2)	6(2)	7(2)	-5(2)
C(6')	33(2)	34(2)	47(2)	2(2)	-1(2)	-12(2)
C(7')	33(2)	29(2)	38(2)	-4(2)	-2(2)	-4(2)
C(8')	28(2)	31(2)	29(2)	-3(1)	3(1)	1(2)
C(9')	26(2)	28(2)	24(2)	-5(1)	1(1)	8(1)
C(10')	27(2)	29(2)	28(2)	-9(1)	-3(1)	10(1)
C(11')	30(2)	28(2)	42(2)	-14(2)	-6(2)	6(2)
C(12')	27(2)	30(2)	47(2)	-4(2)	5(2)	-3(2)
C(13')	27(2)	30(2)	30(2)	-5(1)	5(1)	0(1)
C(14')	29(2)	45(2)	24(2)	6(1)	5(1)	15(2)
C(15')	34(2)	66(3)	29(2)	-6(2)	5(2)	7(2)
C(16')	39(2)	81(3)	29(2)	-10(2)	7(2)	6(2)
C(17')	40(2)	68(3)	25(2)	7(2)	12(2)	22(2)
C(18')	45(2)	48(2)	35(2)	15(2)	18(2)	16(2)
Cl(1')	30(1)	37(1)	30(1)	-4(1)	8(1)	2(1)

Cl(2')	39(1)	31(1)	25(1)	-2(1)	6(1)	-1(1)
O	28(1)	27(1)	26(1)	-2(1)	9(1)	-3(1)
O(1)	35(1)	34(1)	30(1)	1(1)	8(1)	-2(1)
O(2')	22(5)	177(15)	28(5)	-14(6)	-7(4)	33(7)
O(2)	215(9)	162(8)	123(6)	-79(5)	141(6)	-142(7)

Table A2.116. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for $[Fe(tpt)Cl_2]_2(\mu\text{-}O).2(H_2O)$, **6.4.**

	x	y	z	U(eq)
H(5)	3904	1418	9682	31
H(6)	5527	509	9454	35
H(7)	6363	620	8529	35
H(8)	5588	1620	7852	31
H(10)	67	4988	8357	33
H(11)	-200	5752	7473	40
H(12)	928	5363	6665	41
H(13)	2335	4262	6764	32
H(15)	1448	2175	10389	36
H(16)	416	2212	11259	48
H(17)	-1008	3283	11405	44
H(18)	-1351	4305	10689	48
H(5')	8465	4496	5532	41
H(6')	8633	5574	6241	46
H(7')	7401	5515	7058	40
H(8')	6013	4411	7134	35
H(10')	4461	744	4709	34
H(11')	2865	-67	5075	40
H(12')	2155	259	6004	41
H(13')	3005	1394	6531	35
H(15')	6750	1395	3774	52
H(16')	7686	1339	2868	59
H(17')	9333	2233	2709	53
H(18')	10002	3163	3445	50
H(1A)	3301	5853	8678	50
H(1B)	3820	4855	8687	50

Table 7. Hydrogen bonds for 9RZGR(sad) [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1A)...Cl(1')#1	1.05	2.15	3.197(3)	176.1
O(1)-H(1B)...Cl(2)	1.05	2.11	3.148(3)	171.4

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y+1/2,-z+3/2

Table A2.117. Crystal data and structure refinement for $[Cu(OH_2)_6](NO_3)_2$, **6.5.**

Identification code	9rz12
Empirical formula	Cu H12 N2 O12
Formula weight	295.66
Temperature	93(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, *P* -1
Unit cell dimensions	a = 5.7404(8) Å alpha = 106.428(2)°. b = 7.6452(10) Å beta = 98.399(2)°. c = 11.4655(15) Å gamma = 101.504(2)°.
Volume	461.84(11) $Å^3$
Z, Calculated density	2, 2.126 Mg/m^3
Absorption coefficient	2.432 mm^{-1}
F(000)	302
Crystal size	0.55 x 0.34 x 0.12 mm
Theta range for data collection	3.71 to 25.05°.
Limiting indices	-6<=h<=6, -8<=k<=8, -13<=l<=13
Reflections collected / unique	2917 / 1556 [R(int) = 0.0195]
Completeness to theta = 25.00	95.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.74382 and 0.341138
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1556 / 18 / 172
Goodness-of-fit on F^2	0.907
Final R indices [I>2sigma(I)]	R1 = 0.0343, wR2 = 0.0952
R indices (all data)	R1 = 0.0350, wR2 = 0.0957
Largest diff. peak and hole	0.598 and -1.142 $e.A^{-3}$

Table A2.118. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for $[Cu(OH_2)_6](NO_3)_2$**, 6.5.** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Cu	1724(1)	1452(1)	2324(1)	10(1)
O(1)	2520(4)	-653(4)	1031(2)	10(1)
O(2)	5425(4)	2400(4)	3146(2)	9(1)
O(3)	978(4)	3582(4)	3683(2)	10(1)
O(4)	-1905(4)	364(3)	1453(2)	9(1)
O(5)	2220(4)	3095(4)	1238(2)	11(1)
O(6)	1346(4)	-50(4)	3531(2)	10(1)
N(1)	7929(5)	5522(4)	1275(3)	9(1)
O(11)	6585(4)	6598(4)	1232(2)	12(1)
O(12)	7080(4)	3869(4)	1234(2)	12(1)
O(13)	10202(4)	6086(4)	1371(2)	13(1)
N(2)	5813(5)	7634(4)	3903(3)	10(1)
O(21)	6533(5)	9126(4)	3697(2)	12(1)
O(22)	3683(4)	7234(4)	4107(2)	14(1)
O(23)	7148(4)	6530(4)	3925(2)	13(1)

Table A2.119. Selected bond lengths [Å] and angles [°] for $[Cu(OH_2)_6](NO_3)_2$, **6.5.**

Cu–O(5)	2.014(2)
Cu–O(1)	2.034(2)
Cu–O(6)	2.041(2)
Cu–O(4)	2.064(2)
Cu–O(3)	2.074(2)
Cu–O(2)	2.084(2)
O(5)–Cu–O(1)	89.49(10)
O(5)–Cu–O(6)	175.94(10)
O(1)–Cu–O(6)	93.58(10)
O(5)–Cu–O(4)	91.38(10)
O(1)–Cu–O(4)	88.81(10)
O(6)–Cu–O(4)	91.34(10)
O(5)–Cu–O(3)	91.72(10)
O(1)–Cu–O(3)	178.31(10)
O(6)–Cu–O(3)	85.17(10)
O(4)–Cu–O(3)	92.35(10)
O(5)–Cu–O(2)	89.50(10)
O(1)–Cu–O(2)	87.93(10)
O(6)–Cu–O(2)	87.96(10)
O(4)–Cu–O(2)	176.61(9)
O(3)–Cu–O(2)	90.89(10)

Symmetry transformations used to generate equivalent atoms:

Table A2.120. Bond lengths [Å] and angles [°] for $[Cu(OH_2)_6](NO_3)_2$, **6.5.**

Cu–O(5)	2.014(2)
Cu–O(1)	2.034(2)
Cu–O(6)	2.041(2)
Cu–O(4)	2.064(2)
Cu–O(3)	2.074(2)
Cu–O(2)	2.084(2)
O(1)–H(1A)	0.981(18)
O(1)–H(1B)	0.966(18)
O(2)–H(2A)	0.976(18)
O(2)–H(2B)	0.981(18)
O(3)–H(3A)	0.977(18)
O(3)–H(3B)	0.966(18)
O(4)–H(4A)	0.970(18)
O(4)–H(4B)	0.974(18)
O(5)–H(5A)	0.974(18)
O(5)–H(5B)	0.963(18)
O(6)–H(6A)	0.973(18)
O(6)–H(6B)	0.978(18)
N(1)–O(11)	1.241(4)
N(1)–O(12)	1.245(4)
N(1)–O(13)	1.268(4)
N(2)–O(21)	1.233(4)
N(2)–O(23)	1.252(4)
N(2)–O(22)	1.272(4)
O(5)–Cu–O(1)	89.49(10)
O(5)–Cu–O(6)	175.94(10)
O(1)–Cu–O(6)	93.58(10)
O(5)–Cu–O(4)	91.38(10)
O(1)–Cu–O(4)	88.81(10)
O(6)–Cu–O(4)	91.34(10)
O(5)–Cu–O(3)	91.72(10)
O(1)–Cu–O(3)	178.31(10)
O(6)–Cu–O(3)	85.17(10)
O(4)–Cu–O(3)	92.35(10)
O(5)–Cu–O(2)	89.50(10)
O(1)–Cu–O(2)	87.93(10)
O(6)–Cu–O(2)	87.96(10)
O(4)–Cu–O(2)	176.61(9)
O(3)–Cu–O(2)	90.89(10)
Cu–O(1)–H(1A)	116(2)
Cu–O(1)–H(1B)	113(2)
H(1A)–O(1)–H(1B)	113(2)
Cu–O(2)–H(2A)	112(2)
Cu–O(2)–H(2B)	113(2)
H(2A)–O(2)–H(2B)	112(2)
Cu–O(3)–H(3A)	108(2)
Cu–O(3)–H(3B)	110(2)
H(3A)–O(3)–H(3B)	113(2)
Cu–O(4)–H(4A)	111(2)
Cu–O(4)–H(4B)	108(2)
H(4A)–O(4)–H(4B)	113(2)
Cu–O(5)–H(5A)	114(2)
Cu–O(5)–H(5B)	118(2)
H(5A)–O(5)–H(5B)	114(3)
Cu–O(6)–H(6A)	110(2)

Cu-O(6)-H(6B)	122(2)
H(6A)-O(6)-H(6B)	112(2)
O(11)-N(1)-O(12)	120.8(3)
O(11)-N(1)-O(13)	120.7(3)
O(12)-N(1)-O(13)	118.5(3)
O(21)-N(2)-O(23)	121.1(3)
O(21)-N(2)-O(22)	118.9(3)
O(23)-N(2)-O(22)	120.0(3)

Symmetry transformations used to generate equivalent atoms:

Table A2.121. Anisotropic displacement parameters (A^2 x 10^3) for $[Cu(OH_2)_6](NO_3)_2$**, 6.5.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Cu	6(1)	11(1)	13(1)	3(1)	4(1)	1(1)
O(1)	8(1)	8(1)	12(1)	2(1)	5(1)	1(1)
O(2)	5(1)	10(1)	13(1)	5(1)	2(1)	0(1)
O(3)	9(1)	10(1)	12(1)	3(1)	5(1)	3(1)
O(4)	5(1)	8(1)	13(1)	3(1)	4(1)	1(1)
O(5)	7(1)	12(1)	16(1)	8(1)	6(1)	2(1)
O(6)	6(1)	11(1)	14(1)	6(1)	5(1)	1(1)
N(1)	8(1)	11(2)	8(1)	2(1)	3(1)	1(1)
O(11)	10(1)	12(1)	15(1)	4(1)	5(1)	4(1)
O(12)	10(1)	8(1)	22(1)	7(1)	7(1)	1(1)
O(13)	5(1)	12(1)	22(1)	6(1)	5(1)	-1(1)
N(2)	8(1)	12(2)	9(1)	1(1)	3(1)	2(1)
O(21)	12(1)	11(1)	18(1)	8(1)	8(1)	2(1)
O(22)	7(1)	14(2)	23(1)	5(1)	7(1)	0(1)
O(23)	11(1)	14(1)	14(1)	4(1)	4(1)	5(1)

Table A2.122. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for $[Cu(OH_2)_6](NO_3)_2$, **6.5.**

	x	y	z	U(eq)
H(1A)	2300(70)	−580(50)	180(20)	11
H(1B)	1800(70)	−1890(40)	1060(30)	11
H(2A)	6070(70)	3720(30)	3230(30)	11
H(2B)	5810(70)	2160(50)	3940(20)	11
H(3A)	1850(70)	4790(40)	3650(30)	12
H(3B)	1380(70)	3430(50)	4490(20)	12
H(4A)	−2910(60)	1070(40)	1900(30)	11
H(4B)	−2340(70)	−970(30)	1370(40)	11
H(5A)	3880(40)	3400(50)	1110(40)	13
H(5B)	1440(60)	4120(40)	1340(40)	13
H(6A)	−370(40)	−480(50)	3520(40)	12
H(6B)	2210(60)	−1020(50)	3560(40)	12

Table A2.123. Hydrogen bonds for $[Cu(OH_2)_6](NO_3)_2$, **6.5** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1A)...O(4)#1	0.981(18)	1.914(18)	2.894(3)	179(3)
O(1)-H(1B)...O(13)#2	0.966(18)	1.788(19)	2.741(4)	168(4)
O(2)-H(2B)...O(22)#3	0.981(18)	2.12(2)	3.038(4)	156(3)
O(2)-H(2A)...O(23)	0.976(18)	2.00(2)	2.940(4)	162(3)
O(2)-H(2B)...O(21)#4	0.981(18)	2.38(3)	2.912(4)	113(3)
O(3)-H(3A)...O(22)	0.977(18)	1.83(2)	2.779(4)	162(3)
O(3)-H(3B)...O(23)#3	0.966(18)	1.88(2)	2.827(4)	167(3)
O(4)-H(4A)...O(2)#5	0.970(18)	1.99(2)	2.942(4)	167(4)
O(4)-H(4A)...O(1)#5	0.970(18)	2.60(4)	3.070(3)	110(3)
O(4)-H(4B)...O(11)#2	0.974(18)	1.79(2)	2.763(4)	175(3)
O(5)-H(5A)...O(12)	0.974(18)	1.78(2)	2.735(3)	166(4)
O(5)-H(5A)...N(1)	0.974(18)	2.50(3)	3.417(4)	156(3)
O(5)-H(5A)...O(11)	0.974(18)	2.58(3)	3.285(3)	130(3)
O(5)-H(5B)...O(13)#5	0.963(18)	1.78(2)	2.740(4)	172(4)
O(5)-H(5B)...N(1)#5	0.963(18)	2.47(2)	3.365(4)	155(3)
O(5)-H(5B)...O(12)#5	0.963(18)	2.45(3)	3.123(3)	126(3)
O(6)-H(6B)...O(21)#4	0.978(18)	2.44(3)	3.154(3)	130(3)
O(6)-H(6B)...O(22)#4	0.978(18)	1.91(2)	2.860(4)	162(4)
O(6)-H(6B)...N(2)#4	0.978(18)	2.51(2)	3.436(4)	157(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y,-z #2 x-1,y-1,z #3 -x+1,-y+1,-z+1
#4 x,y-1,z #5 x-1,y,z

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Appendix 3

Crystallographic Information Files (CIFs)

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C13 C 0.1583(3) 0.3396(3) 0.2837(3) 0.0381(12) Uani 1 1 d . . .
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Cl2 Cl 0.45646(18) 0.1183(2) 0.3307(3) 0.205(2) Uani 1 1 d . . .
O4 O 0.4061(2) 0.1595(2) 0.5370(2) 0.0393(8) Uani 1 1 d D . .
H4A H 0.432(3) 0.117(3) 0.514(3) 0.059 Uiso 1 1 d D . .
H4B H 0.441(2) 0.182(4) 0.566(2) 0.059 Uiso 1 1 d D . .
O5 O 0.2702(5) 0.3712(3) 0.4530(3) 0.107(2) Uani 1 1 d D . .
H5A H 0.283(6) 0.419(4) 0.431(4) 0.161 Uiso 1 1 d D . .
H5B H 0.290(6) 0.379(6) 0.496(2) 0.161 Uiso 1 1 d D . .
O6 O 0.5755(5) 0.0407(9) 0.4151(5) 0.176(4) Uani 1 1 d D . .
H6A H 0.548(8) 0.000(8) 0.434(7) 0.265 Uiso 1 1 d D . .
H6B H 0.563(9) 0.043(12) 0.371(2) 0.265 Uiso 1 1 d D . .

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N1 0.033(2) 0.0298(19) 0.032(2) -0.0004(17) 0.0078(18) -0.0015(17)
N2 0.030(2) 0.0317(19) 0.030(2) -0.0047(17) 0.0016(17) 0.0005(17)
N3 0.036(2) 0.0264(19) 0.035(2) -0.0043(17) 0.0045(18) -0.0012(17)
N4 0.040(2) 0.0300(19) 0.030(2) -0.0057(17) 0.0004(18) -0.0003(18)
N5 0.038(2) 0.0271(19) 0.034(2) -0.0005(17) 0.0060(18) -0.0022(17)
N6 0.040(2) 0.037(2) 0.031(2) -0.0058(18) 0.0031(19) -0.0066(19)
O1 0.057(2) 0.0305(18) 0.042(2) -0.0004(16) 0.0087(18) 0.0022(16)
O2 0.038(2) 0.054(2) 0.040(2) -0.0009(17) 0.0024(16) -0.0090(17)
O3 0.070(3) 0.045(2) 0.047(2) -0.0037(19) -0.003(2) -0.008(2)
C1 0.032(2) 0.031(2) 0.030(3) -0.0009(19) 0.009(2) 0.002(2)
C2 0.032(2) 0.029(2) 0.031(3) -0.0056(19) 0.004(2) -0.0009(19)
C3 0.033(3) 0.025(2) 0.035(3) -0.001(2) 0.009(2) 0.0017(19)
C4 0.033(3) 0.031(2) 0.037(3) -0.001(2) 0.011(2) 0.000(2)
C5 0.041(3) 0.030(2) 0.045(3) -0.004(2) 0.008(2) 0.002(2)
C6 0.051(3) 0.033(3) 0.045(3) 0.003(2) 0.007(3) 0.009(2)
C7 0.054(3) 0.046(3) 0.038(3) 0.003(2) -0.002(3) 0.014(3)
C8 0.042(3) 0.041(3) 0.033(3) -0.005(2) -0.003(2) 0.006(2)
C9 0.033(2) 0.026(2) 0.032(3) -0.0011(19) 0.007(2) -0.003(2)
C10 0.035(3) 0.031(2) 0.038(3) 0.003(2) 0.007(2) 0.000(2)
C11 0.048(3) 0.032(3) 0.043(3) 0.004(2) 0.010(2) 0.003(2)
C12 0.048(3) 0.022(2) 0.050(3) 0.001(2) 0.010(3) 0.000(2)
C13 0.048(3) 0.029(2) 0.037(3) -0.006(2) 0.005(2) -0.008(2)
C14 0.035(3) 0.031(2) 0.037(3) -0.006(2) 0.008(2) -0.001(2)
C15 0.048(3) 0.039(3) 0.063(4) -0.019(3) -0.009(3) 0.009(2)
C16 0.061(4) 0.043(3) 0.076(5) -0.030(3) -0.008(3) 0.005(3)
C17 0.048(3) 0.051(3) 0.049(3) -0.022(3) 0.002(3) -0.006(3)
C18 0.041(3) 0.044(3) 0.035(3) -0.008(2) -0.001(2) -0.004(2)
Cl1 0.0510(8) 0.0549(8) 0.0406(8) -0.0074(6) 0.0015(6) 0.0026(6)
Cl2 0.104(2) 0.126(2) 0.372(6) -0.074(3) -0.059(3) 0.0076(18)
O4 0.038(2) 0.0354(19) 0.045(2) -0.0022(16) 0.0074(17) -0.0048(15)
O5 0.207(7) 0.057(3) 0.051(3) -0.003(2) -0.028(4) -0.004(4)
O6 0.114(6) 0.253(11) 0.172(8) -0.060(8) 0.068(6) -0.067(6)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Ni O1 2.063(4) . ?
Ni O3 2.073(4) . ?
Ni N5 2.160(4) . ?
Ni N4 2.162(4) . ?
N1 C1 1.331(6) . ?
N1 C3 1.340(6) . ?
N2 C1 1.328(6) . ?
N2 C2 1.352(6) . ?
N3 C3 1.319(6) . ?
N3 C2 1.354(6) . ?
N4 C8 1.333(6) . ?
N4 C4 1.350(6) . ?
N5 C13 1.337(6) . ?
N5 C9 1.364(6) . ?
N6 C18 1.332(6) . ?
N6 C14 1.333(6) . ?
O1 H1A 0.831(18) . ?
O1 H1B 0.810(18) . ?
O2 H2A 0.812(18) . ?
O2 H2B 0.817(18) . ?
O3 H3A 0.809(17) . ?
O3 H3B 0.791(17) . ?
C1 C9 1.482(6) . ?
C2 C14 1.469(7) . ?
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C5 C6 1.396(7) . ?
C5 H5 0.9300 . ?
C6 C7 1.356(7) . ?
C6 H6 0.9300 . ?
C7 C8 1.383(7) . ?
C7 H7 0.9300 . ?
C8 H8 0.9300 . ?
C9 C10 1.359(6) . ?
C10 C11 1.400(6) . ?
C10 H10 0.9300 . ?
C11 C12 1.377(7) . ?
C11 H11 0.9300 . ?
C12 C13 1.365(7) . ?
C12 H12 0.9300 . ?
C13 H13 0.9300 . ?
C14 C15 1.383(6) . ?
C15 C16 1.367(8) . ?
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C15 H15 0.9300 . ?
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C18 H18 0.9300 . ?
O4 H4A 0.903(14) . ?
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O1 Ni O3 90.13(16) . . ?
N1 Ni N5 76.79(15) . . ?
O2 Ni N5 88.60(14) . . ?
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N1 Ni N4 76.82(15) . . ?
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O3 Ni N4 101.14(16) . . ?
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C1 N1 C3 117.7(4) . . ?
C1 N1 Ni 121.4(3) . . ?
C3 N1 Ni 120.8(3) . . ?
C1 N2 C2 114.8(4) . . ?
C3 N3 C2 115.1(4) . . ?
C8 N4 C4 116.2(4) . . ?
C8 N4 Ni 129.5(3) . . ?
C4 N4 Ni 114.2(3) . . ?
C13 N5 C9 116.4(4) . . ?
C13 N5 Ni 129.6(3) . . ?
C9 N5 Ni 114.0(3) . . ?
C18 N6 C14 117.6(4) . . ?
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Ni O1 H1B 109.7(19) . . ?
H1A O1 H1B 113(3) . . ?
Ni O2 H2A 111.3(19) . . ?
Ni O2 H2B 111.9(19) . . ?
H2A O2 H2B 114(3) . . ?
Ni O3 H3A 109.0(18) . . ?
Ni O3 H3B 110.4(19) . . ?
H3A O3 H3B 117(3) . . ?
N2 C1 N1 123.8(4) . . ?
N2 C1 C9 122.0(4) . . ?
N1 C1 C9 114.2(4) . . ?
N2 C2 N3 125.0(4) . . ?
N2 C2 C14 116.9(4) . . ?
N3 C2 C14 118.1(4) . . ?
N3 C3 N1 123.6(4) . . ?
N3 C3 C4 122.3(4) . . ?
N1 C3 C4 114.2(4) . . ?
N4 C4 C5 123.5(5) . . ?
N4 C4 C3 113.8(4) . . ?
C5 C4 C3 122.6(4) . . ?
C4 C5 C6 119.2(5) . . ?
C4 C5 H5 120.4 . . ?
C6 C5 H5 120.4 . . ?
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C7 C6 H6 121.3 . . ?
C5 C6 H6 121.3 . . ?
C6 C7 C8 120.3(5) . . ?
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C8 C7 H7 119.8 . . ?
N4 C8 C7 123.2(5) . . ?
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C7 C8 H8 118.4 . . ?
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N5 C9 C1 113.5(4) . . ?
C9 C10 C11 117.7(5) . . ?
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C12 C11 H11 120.5 . . ?
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C11 C12 H12 120.3 . . ?
N5 C13 C12 123.5(5) . . ?
N5 C13 H13 118.3 . . ?
C12 C13 H13 118.3 . . ?
N6 C14 C15 122.9(5) . . ?

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N6 C14 C2 116.9(4) . . ?
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C15 C16 C17 119.4(5) . . ?
C15 C16 H16 120.3 . . ?
C17 C16 H16 120.3 . . ?
C18 C17 C16 118.0(5) . . ?
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C16 C17 H17 121.0 . . ?
N6 C18 C17 123.4(5) . . ?
N6 C18 H18 118.3 . . ?
C17 C18 H18 118.3 . . ?
H4A O4 H4B 104(3) . . ?
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O1 Ni N1 C1 92.5(3) . . . . ?
N5 Ni N1 C1 2.9(3) . . . . ?
N4 Ni N1 C1 -178.9(4) . . . . ?
O2 Ni N1 C3 90.4(3) . . . . ?
O1 Ni N1 C3 -91.9(3) . . . . ?
N5 Ni N1 C3 178.5(4) . . . . ?
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O2 Ni N4 C8 92.4(4) . . . . ?
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O3 Ni N4 C8 7.1(4) . . . . ?
N5 Ni N4 C8 -172.6(4) . . . . ?
N1 Ni N4 C4 1.1(3) . . . . ?
O2 Ni N4 C4 -90.1(3) . . . . ?
O1 Ni N4 C4 94.6(3) . . . . ?
O3 Ni N4 C4 -175.3(3) . . . . ?
N5 Ni N4 C4 5.0(5) . . . . ?
N1 Ni N5 C13 179.6(4) . . . . ?
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O3 Ni N5 C9 173.3(3) . . . . ?
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C2 N2 C1 N1 -2.4(6) . . . . ?
C2 N2 C1 C9 176.4(4) . . . . ?
C3 N1 C1 N2 1.1(6) . . . . ?
Ni N1 C1 N2 176.8(3) . . . . ?
C3 N1 C1 C9 -177.9(4) . . . . ?
Ni N1 C1 C9 -2.1(5) . . . . ?
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C2 N3 C3 N1 0.1(6) . . . . ?
C2 N3 C3 C4 179.8(4) . . . . ?
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Ni N1 C3 N3 -175.6(3) . . . . ?
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Ni N1 C3 C4 4.7(5) . . . . ?
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Ni N4 C4 C5 -177.5(4) . . . . ?
C8 N4 C4 C3 178.8(4) . . . . ?
Ni N4 C4 C3 0.8(5) . . . . ?
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N1 C3 C4 N4 -3.4(6) . . . . ?
N3 C3 C4 C5 -4.7(7) . . . . ?
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Ni N4 C8 C7 175.9(4) . . . . ?
C6 C7 C8 N4 1.6(8) . . . . ?
C13 N5 C9 C10 0.5(7) . . . . ?
Ni N5 C9 C10 -177.1(4) . . . . ?
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N1 C1 C9 C10 179.3(4) . . . . ?
N2 C1 C9 N5 -179.8(4) . . . . ?
N1 C1 C9 N5 -0.8(5) . . . . ?
N5 C9 C10 C11 -0.9(7) . . . . ?

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C9 C10 C11 C12 0.3(7) . . . . ?
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C18 N6 C14 C15 -1.4(7) . . . . ?
C18 N6 C14 C2 177.0(4) . . . . ?
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N2 C2 C14 C15 168.6(5) . . . . ?
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N6 C14 C15 C16 1.0(8) . . . . ?
C2 C14 C15 C16 -177.3(5) . . . . ?
C14 C15 C16 C17 1.7(9) . . . . ?
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O2 H2A Cl2  0.812(18) 2.07(2) 2.874(5) 174(5) .
O2 H2B Cl1  0.817(18) 2.24(2) 3.031(4) 162(5) .
O3 H3A O5  0.809(17) 1.85(2) 2.621(7) 158(3) .
O3 H3B O4  0.791(17) 2.38(2) 3.031(6) 140(3) .
O3 H3B O5  0.791(17) 2.58(4) 3.213(8) 138(4) 7_556
O5 H5A Cl1  0.86(2) 2.29(5) 3.075(5) 152(10) 8_556
O5 H5B O1  0.86(2) 2.07(5) 2.883(7) 157(10) 7_556
O6 H6A O4  0.85(2) 2.51(14) 3.081(14) 125(14) 5_656
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'R.M.Hartshorn'
'R.Zibaseresht'

_journal_name_full         'Aust. J. Chem.'

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'-x, -y, -z'

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_diffrn_radiation_source         'fine-focus sealed tube'
_diffrn_radiation_monochromator  graphite
_diffrn_measurement_device_type  'SMART/CCD'
_diffrn_measurement_method       'phi and omega scan'
_diffrn_detector_area_resol_mean  8.192
_diffrn_standards_number         ?
_diffrn_standards_interval_count  ?
_diffrn_standards_interval_time  ?
_diffrn_standards_decay_%        ?
_diffrn_reflns_number            20130
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_diffrn_reflns_av_R_equivalents   0.0367
_diffrn_reflns_av_sigmaI/netI     0.0564
_diffrn_reflns_limit_h_min        -11
_diffrn_reflns_limit_h_max        16
_diffrn_reflns_limit_k_min        -16
_diffrn_reflns_limit_k_max        16
_diffrn_reflns_limit_l_min        -13
_diffrn_reflns_limit_l_max        17
_diffrn_reflns_theta_min          1.63
_diffrn_reflns_theta_max          26.40
_reflns_number_total              8363
_reflns_number_gt                 5944
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        'Bruker SMART'
_computing_cell_refinement        'Bruker SAINT+'
_computing_data_reduction         'Bruker XPREP'
_computing_structure_solution     'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics     'Bruker SHELXTL'
_computing_publication_material   'Bruker SHELXTL'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0414P)^2^+0.0313P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          8363
_refine_ls_number_parameters      586
_refine_ls_number_restraints      5
_refine_ls_R_factor_all           0.0674
_refine_ls_R_factor_gt            0.0360
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_refine_ls_wR_factor_ref          0.0856
_refine_ls_wR_factor_gt           0.0772
_refine_ls_goodness_of_fit_ref    1.014
_refine_ls_restrained_S_all       1.015
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Ni1 Ni 0.21412(3) 0.59257(3) 0.24235(3) 0.02182(9) Uani 1 1 d . . .
N1 N 0.38258(18) 0.49410(16) 0.20062(16) 0.0210(5) Uani 1 1 d . . .
N2 N 0.58527(17) 0.45972(16) 0.10184(15) 0.0202(5) Uani 1 1 d . . .
N3 N 0.51988(18) 0.32260(16) 0.20006(16) 0.0208(5) Uani 1 1 d . . .
N4 N 0.19863(18) 0.44812(17) 0.31450(16) 0.0220(5) Uani 1 1 d . . .
N5 N 0.31239(18) 0.68578(16) 0.14373(16) 0.0221(5) Uani 1 1 d . . .
N6 N 0.81331(19) 0.32235(17) 0.03787(17) 0.0268(5) Uani 1 1 d . . .
Cl1 Cl 0.22210(6) 0.66089(5) 0.39702(5) 0.02939(16) Uani 1 1 d . . .
Cl2 Cl 0.02011(6) 0.70726(6) 0.28011(6) 0.03666(19) Uani 1 1 d . . .
O1 O 0.21896(18) 0.51929(15) 0.11167(15) 0.0289(5) Uani 1 1 d . . .
H1A H 0.2121 0.5635 0.0683 0.043 Uiso 1 1 calc R . .
H1B H 0.182(3) 0.483(2) 0.122(2) 0.043 Uiso 1 1 d . . .
C1 C 0.4716(2) 0.5237(2) 0.13629(18) 0.0194(6) Uani 1 1 d . . .
C2 C 0.6051(2) 0.3604(2) 0.13803(18) 0.0192(6) Uani 1 1 d . . .
C3 C 0.4094(2) 0.39274(19) 0.22778(19) 0.0188(5) Uani 1 1 d . . .
C4 C 0.3031(2) 0.3646(2) 0.29089(19) 0.0205(6) Uani 1 1 d . . .
C5 C 0.3103(2) 0.2626(2) 0.3218(2) 0.0288(7) Uani 1 1 d . . .
H5 H 0.3834 0.2068 0.3039 0.035 Uiso 1 1 calc R . .
C6 C 0.2050(3) 0.2459(2) 0.3806(2) 0.0353(7) Uani 1 1 d . . .
H6 H 0.2066 0.1779 0.4020 0.042 Uiso 1 1 calc R . .
C7 C 0.0984(2) 0.3304(2) 0.4071(2) 0.0320(7) Uani 1 1 d . . .
H7 H 0.0275 0.3207 0.4479 0.038 Uiso 1 1 calc R . .
C8 C 0.0986(2) 0.4297(2) 0.3723(2) 0.0262(6) Uani 1 1 d . . .
H8 H 0.0262 0.4864 0.3895 0.031 Uiso 1 1 calc R . .
C9 C 0.4318(2) 0.6351(2) 0.10424(19) 0.0202(6) Uani 1 1 d . . .
C10 C 0.5102(2) 0.6812(2) 0.0380(2) 0.0236(6) Uani 1 1 d . . .
H10 H 0.5918 0.6434 0.0115 0.028 Uiso 1 1 calc R . .
C11 C 0.4632(3) 0.7862(2) 0.0122(2) 0.0296(7) Uani 1 1 d . . .
```

H11 H 0.5133 0.8202 -0.0314 0.036 Uiso 1 1 calc R . .
C12 C 0.3422(3) 0.8386(2) 0.0521(2) 0.0291(7) Uani 1 1 d . . .
H12 H 0.3094 0.9085 0.0355 0.035 Uiso 1 1 calc R . .
C13 C 0.2690(2) 0.7866(2) 0.1174(2) 0.0252(6) Uani 1 1 d . . .
H13 H 0.1870 0.8228 0.1439 0.030 Uiso 1 1 calc R . .
C14 C 0.7305(2) 0.2863(2) 0.10649(19) 0.0203(6) Uani 1 1 d . . .
C15 C 0.7585(2) 0.1859(2) 0.1455(2) 0.0258(6) Uani 1 1 d . . .
H15 H 0.6987 0.1633 0.1917 0.031 Uiso 1 1 calc R . .
C16 C 0.8763(2) 0.1197(2) 0.1151(2) 0.0325(7) Uani 1 1 d . . .
H16 H 0.8973 0.0519 0.1402 0.039 Uiso 1 1 calc R . .
C17 C 0.9625(3) 0.1563(2) 0.0465(2) 0.0376(8) Uani 1 1 d . . .
H17 H 1.0428 0.1144 0.0255 0.045 Uiso 1 1 calc R . .
C18 C 0.9261(2) 0.2570(2) 0.0096(2) 0.0345(7) Uani 1 1 d . . .
H18 H 0.9844 0.2803 -0.0380 0.041 Uiso 1 1 calc R . .
Ni1' Ni 0.35690(3) 0.87251(3) 0.50375(3) 0.02075(9) Uani 1 1 d . . .
N1' N 0.32219(18) 0.74920(16) 0.58043(16) 0.0201(5) Uani 1 1 d . . .
N2' N 0.39393(18) 0.57887(16) 0.63934(16) 0.0225(5) Uani 1 1 d . . .
N3' N 0.18318(18) 0.67444(16) 0.69172(16) 0.0215(5) Uani 1 1 d . . .
N4' N 0.16415(18) 0.93090(16) 0.57835(16) 0.0226(5) Uani 1 1 d . . .
N5' N 0.53142(18) 0.75435(17) 0.47245(16) 0.0226(5) Uani 1 1 d . . .
N6' N 0.1438(2) 0.50980(17) 0.81184(18) 0.0299(6) Uani 1 1 d . . .
Cl1' Cl 0.38428(6) 0.94634(5) 0.63735(5) 0.03025(17) Uani 1 1 d . . .
O1' O 0.3557(2) 0.81558(17) 0.36997(16) 0.0385(5) Uani 1 1 d . . .
H1A' H 0.3689 0.8567 0.3239 0.058 Uiso 1 1 calc R . .
H1B' H 0.328(3) 0.773(3) 0.366(3) 0.058 Uiso 1 1 d . . .
O2' O 0.38207(17) 1.00257(15) 0.42285(14) 0.0267(4) Uani 1 1 d D . .
H2A' H 0.4378 1.0134 0.4285 0.040 Uiso 1 1 calc RD . .
H2B' H 0.393(3) 0.998(2) 0.3648(14) 0.040 Uiso 1 1 d D . .
C1' C 0.4101(2) 0.6613(2) 0.58374(19) 0.0215(6) Uani 1 1 d . . .
C2' C 0.2785(2) 0.5891(2) 0.69210(19) 0.0210(6) Uani 1 1 d . . .
C3' C 0.2106(2) 0.7529(2) 0.63557(19) 0.0206(6) Uani 1 1 d . . .
C4' C 0.1180(2) 0.8576(2) 0.6324(2) 0.0220(6) Uani 1 1 d . . .
C5' C -0.0015(2) 0.8783(2) 0.6845(2) 0.0272(6) Uani 1 1 d . . .
H5' H -0.0289 0.8253 0.7203 0.033 Uiso 1 1 calc R . .
C6' C -0.0809(2) 0.9802(2) 0.6825(2) 0.0328(7) Uani 1 1 d . . .
H6' H -0.1626 0.9966 0.7158 0.039 Uiso 1 1 calc R . .
C7' C -0.0353(2) 1.0570(2) 0.6297(2) 0.0326(7) Uani 1 1 d . . .
H7' H -0.0859 1.1263 0.6283 0.039 Uiso 1 1 calc R . .
C8' C 0.0865(2) 1.0287(2) 0.5791(2) 0.0272(6) Uani 1 1 d . . .
H8' H 0.1160 1.0807 0.5438 0.033 Uiso 1 1 calc R . .
C9' C 0.5310(2) 0.6626(2) 0.52154(19) 0.0212(6) Uani 1 1 d . . .
C10' C 0.6325(2) 0.5788(2) 0.5160(2) 0.0254(6) Uani 1 1 d . . .
H10' H 0.6282 0.5174 0.5510 0.030 Uiso 1 1 calc R . .
C11' C 0.7417(2) 0.5878(2) 0.4569(2) 0.0302(7) Uani 1 1 d . . .
H11' H 0.8120 0.5320 0.4509 0.036 Uiso 1 1 calc R . .
C12' C 0.7444(2) 0.6803(2) 0.4075(2) 0.0331(7) Uani 1 1 d . . .
H12' H 0.8167 0.6884 0.3684 0.040 Uiso 1 1 calc R . .
C13' C 0.6381(2) 0.7615(2) 0.4165(2) 0.0287(6) Uani 1 1 d . . .
H13' H 0.6409 0.8237 0.3823 0.034 Uiso 1 1 calc R . .

C14' C 0.2580(2) 0.4990(2) 0.7558(2) 0.0232(6) Uani 1 1 d . . .
C15' C 0.3533(2) 0.4082(2) 0.7557(2) 0.0249(6) Uani 1 1 d . . .
H15' H 0.4311 0.4032 0.7157 0.030 Uiso 1 1 calc R . .
C16' C 0.3309(3) 0.3252(2) 0.8162(2) 0.0311(7) Uani 1 1 d . . .
H16' H 0.3928 0.2631 0.8169 0.037 Uiso 1 1 calc R . .
C17' C 0.2138(3) 0.3376(2) 0.8753(2) 0.0344(7) Uani 1 1 d . . .
H17' H 0.1955 0.2841 0.9176 0.041 Uiso 1 1 calc R . .
C18' C 0.1250(3) 0.4296(2) 0.8707(2) 0.0395(8) Uani 1 1 d . . .
H18' H 0.0467 0.4365 0.9112 0.047 Uiso 1 1 calc R . .
Cl10 Cl 0.41761(7) 0.97610(7) 0.19945(6) 0.0430(2) Uani 1 1 d . . .
O10 O 0.2428(2) 0.15814(19) 0.11503(18) 0.0511(6) Uani 1 1 d D . .
O11 O 0.1610(2) 0.1129(2) 0.79961(19) 0.0564(7) Uani 1 1 d . . .
O12 O 0.3034(2) 0.0657(2) 0.91988(19) 0.0525(7) Uani 1 1 d . . .
O13 O 0.1028(2) 0.37941(19) 0.1462(2) 0.0467(6) Uani 1 1 d . . .
H10A H 0.283(3) 0.119(3) 0.137(3) 0.070 Uiso 1 1 d D . .
H11A H 0.108(3) 0.159(3) 0.779(3) 0.070 Uiso 1 1 d . . .
H12A H 0.371(3) 0.060(3) 0.877(3) 0.070 Uiso 1 1 d . . .
H13A H 0.036(3) 0.390(3) 0.169(3) 0.070 Uiso 1 1 d . . .
H10B H 0.258(3) 0.139(3) 0.0620(17) 0.070 Uiso 1 1 d D . .
H11B H 0.209(3) 0.080(3) 0.744(3) 0.070 Uiso 1 1 d . . .
H12B H 0.273(3) 0.080(3) 0.879(3) 0.070 Uiso 1 1 d . . .
H13B H 0.142(3) 0.311(3) 0.138(3) 0.070 Uiso 1 1 d . . .

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 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
Ni1 0.01691(18) 0.01926(19) 0.0264(2) 0.00490(14) -0.00485(15) -0.00755(14)
N1 0.0193(11) 0.0188(12) 0.0234(12) 0.0036(9) -0.0058(10) -0.0078(9)
N2 0.0181(12) 0.0219(12) 0.0197(11) 0.0049(9) -0.0044(9) -0.0100(10)
N3 0.0201(12) 0.0191(11) 0.0209(11) 0.0037(9) -0.0051(9) -0.0079(10)
N4 0.0185(12) 0.0242(12) 0.0215(12) 0.0033(9) -0.0053(10) -0.0086(10)
N5 0.0220(12) 0.0191(12) 0.0252(12) 0.0034(9) -0.0085(10) -0.0082(10)
N6 0.0210(12) 0.0247(13) 0.0315(13) 0.0047(10) -0.0052(10) -0.0102(10)
Cl1 0.0349(4) 0.0238(4) 0.0322(4) 0.0042(3) -0.0140(3) -0.0127(3)
Cl2 0.0182(4) 0.0338(4) 0.0511(5) 0.0070(3) -0.0093(3) -0.0067(3)
O1 0.0342(12) 0.0281(11) 0.0287(11) 0.0102(9) -0.0113(10) -0.0183(9)
C1 0.0215(14) 0.0216(14) 0.0172(13) 0.0053(11) -0.0070(11) -0.0115(12)
C2 0.0194(13) 0.0214(14) 0.0166(13) -0.0022(11) -0.0056(11) -0.0075(11)
C3 0.0198(14) 0.0190(14) 0.0176(13) 0.0019(11) -0.0062(11) -0.0082(11)
C4 0.0214(14) 0.0213(14) 0.0177(13) 0.0018(11) -0.0043(11) -0.0100(12)
C5 0.0283(16) 0.0205(15) 0.0331(16) 0.0074(12) -0.0056(13) -0.0114(12)
C6 0.0344(17) 0.0273(16) 0.0412(18) 0.0143(14) -0.0068(15) -0.0184(14)
C7 0.0253(16) 0.0349(17) 0.0363(17) 0.0074(14) -0.0058(13) -0.0186(14)
C8 0.0193(14) 0.0288(16) 0.0260(15) 0.0021(12) -0.0026(12) -0.0103(12)

C9 0.0214(14) 0.0200(14) 0.0188(13) 0.0020(11) -0.0055(11) -0.0093(11)
C10 0.0253(15) 0.0207(14) 0.0256(15) 0.0043(11) -0.0081(12) -0.0115(12)
C11 0.0388(18) 0.0304(16) 0.0248(15) 0.0088(12) -0.0087(13) -0.0232(14)
C12 0.0397(18) 0.0178(14) 0.0324(16) 0.0073(12) -0.0170(14) -0.0106(13)
C13 0.0269(15) 0.0200(14) 0.0304(15) 0.0057(12) -0.0132(13) -0.0085(12)
C14 0.0201(14) 0.0225(14) 0.0183(13) 0.0006(11) -0.0063(11) -0.0085(11)
C15 0.0243(15) 0.0274(16) 0.0228(14) 0.0049(12) -0.0049(12) -0.0109(12)
C16 0.0312(17) 0.0253(16) 0.0334(17) 0.0069(13) -0.0099(14) -0.0050(13)
C17 0.0198(15) 0.0338(18) 0.0467(19) 0.0030(15) -0.0075(14) -0.0012(13)
C18 0.0213(15) 0.0348(18) 0.0401(18) 0.0076(14) -0.0036(13) -0.0116(13)
Ni1' 0.01975(18) 0.01873(18) 0.02422(19) 0.00519(14) -0.00677(15) -0.00988(14)
N1' 0.0184(12) 0.0171(11) 0.0244(12) 0.0026(9) -0.0068(10) -0.0077(9)
N2' 0.0218(12) 0.0188(12) 0.0265(12) 0.0032(10) -0.0067(10) -0.0097(10)
N3' 0.0192(11) 0.0185(11) 0.0262(12) 0.0033(9) -0.0074(10) -0.0078(10)
N4' 0.0220(12) 0.0216(12) 0.0263(12) 0.0073(10) -0.0098(10) -0.0105(10)
N5' 0.0191(12) 0.0229(12) 0.0251(12) 0.0006(10) -0.0050(10) -0.0102(10)
N6' 0.0285(13) 0.0231(13) 0.0344(14) 0.0076(11) -0.0064(11) -0.0120(11)
Cl1' 0.0320(4) 0.0320(4) 0.0278(4) 0.0028(3) -0.0083(3) -0.0160(3)
O1' 0.0572(15) 0.0415(14) 0.0344(12) 0.0112(10) -0.0205(11) -0.0358(12)
O2' 0.0300(11) 0.0264(10) 0.0244(10) 0.0071(9) -0.0108(10) -0.0116(9)
C1' 0.0215(14) 0.0226(14) 0.0211(14) 0.0008(11) -0.0072(12) -0.0093(12)
C2' 0.0226(14) 0.0180(14) 0.0236(14) -0.0012(11) -0.0082(12) -0.0087(11)
C3' 0.0183(14) 0.0197(14) 0.0253(14) 0.0024(11) -0.0077(12) -0.0091(11)
C4' 0.0200(14) 0.0199(14) 0.0263(15) 0.0049(11) -0.0094(12) -0.0072(11)
C5' 0.0208(15) 0.0259(15) 0.0359(17) 0.0061(13) -0.0101(13) -0.0111(12)
C6' 0.0196(15) 0.0312(16) 0.0422(18) 0.0025(14) -0.0099(13) -0.0051(13)
C7' 0.0273(16) 0.0229(15) 0.0442(18) 0.0056(13) -0.0150(14) -0.0042(13)
C8' 0.0292(16) 0.0211(15) 0.0325(16) 0.0081(12) -0.0133(13) -0.0100(12)
C9' 0.0195(14) 0.0228(14) 0.0221(14) 0.0007(11) -0.0066(11) -0.0097(12)
C10' 0.0223(15) 0.0272(15) 0.0263(15) 0.0018(12) -0.0094(12) -0.0084(12)
C11' 0.0189(14) 0.0353(17) 0.0330(16) -0.0011(13) -0.0084(13) -0.0066(13)
C12' 0.0194(15) 0.0444(19) 0.0357(17) -0.0022(14) -0.0040(13) -0.0176(14)
C13' 0.0267(16) 0.0316(16) 0.0280(15) 0.0007(13) -0.0043(13) -0.0169(13)
C14' 0.0263(15) 0.0191(14) 0.0248(14) 0.0029(11) -0.0079(12) -0.0110(12)
C15' 0.0251(15) 0.0242(15) 0.0285(15) 0.0032(12) -0.0127(12) -0.0098(12)
C16' 0.0379(18) 0.0196(15) 0.0374(17) 0.0058(13) -0.0186(14) -0.0084(13)
C17' 0.0416(19) 0.0250(16) 0.0377(18) 0.0140(13) -0.0142(15) -0.0165(14)
C18' 0.0355(18) 0.0336(18) 0.0440(19) 0.0141(15) -0.0055(15) -0.0190(15)
Cl10 0.0486(5) 0.0488(5) 0.0362(4) 0.0067(4) -0.0187(4) -0.0205(4)
O10 0.0515(16) 0.0487(16) 0.0402(14) -0.0013(12) -0.0131(13) -0.0070(12)
O11 0.0502(17) 0.0506(17) 0.0463(15) 0.0035(12) -0.0160(13) 0.0043(13)
O12 0.0547(17) 0.0488(15) 0.0508(17) 0.0110(13) -0.0216(13) -0.0145(14)
O13 0.0340(13) 0.0342(13) 0.0720(17) 0.0076(12) -0.0158(13) -0.0178(12)

_geom_special_details
;
All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles

```
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
;

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Ni1 N1 1.989(2) . ?
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N3 C2 1.353(3) . ?
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C12 H12 0.9300 . ?
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C18' H18' 0.9300 . ?
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O11 H11B 0.82(4) . ?
O12 H12A 0.84(4) . ?
O12 H12B 0.79(4) . ?
O13 H13A 0.76(4) . ?
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C15 C14 C2 120.9(2) . . ?

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Ni1' O2' H2B' 115(2) . . ?
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C4' C5' C6' 118.5(3) . . ?
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C6' C5' H5' 120.7 . . ?
C7' C6' C5' 118.4(3) . . ?
C7' C6' H6' 120.8 . . ?
C5' C6' H6' 120.8 . . ?
C8' C7' C6' 119.1(3) . . ?
C8' C7' H7' 120.5 . . ?
C6' C7' H7' 120.5 . . ?
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C7' C8' H8' 118.2 . . ?
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C9' C10' C11' 118.5(3) . . ?
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N6' C14' C2' 116.6(2) . . ?
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C16' C15' H15' 120.4 . . ?
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C17' C16' H16' 121.0 . . ?
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C18' C17' H17' 120.4 . . ?
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O1 Ni1 N1 C3 -82.77(19) . . . . ?
N4 Ni1 N1 C3 2.81(19) . . . . ?
N5 Ni1 N1 C3 -174.6(2) . . . . ?
Cl1 Ni1 N1 C3 94.12(19) . . . . ?
N1 Ni1 N4 C8 -179.1(2) . . . . ?
O1 Ni1 N4 C8 -93.4(2) . . . . ?
N5 Ni1 N4 C8 -173.5(2) . . . . ?
Cl2 Ni1 N4 C8 -2.4(2) . . . . ?
Cl1 Ni1 N4 C8 90.5(2) . . . . ?
N1 Ni1 N4 C4 -4.72(17) . . . . ?
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N4 Ni1 N5 C9 -5.3(3) . . . . ?
Cl2 Ni1 N5 C9 -176.56(16) . . . . ?
Cl1 Ni1 N5 C9 90.64(17) . . . . ?
C2 N2 C1 N1 -0.5(4) . . . . ?
C2 N2 C1 C9 -179.1(2) . . . . ?
C3 N1 C1 N2 -3.2(4) . . . . ?
Ni1 N1 C1 N2 -176.72(18) . . . . ?
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Ni1 N4 C4 C3 5.8(3) . . . . ?
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C10 C11 C12 C13 0.2(4) . . . . ?
C9 N5 C13 C12 0.0(4) . . . . ?
Ni1 N5 C13 C12 -179.73(19) . . . . ?
C11 C12 C13 N5 0.2(4) . . . . ?
C18 N6 C14 C15 -1.2(4) . . . . ?
C18 N6 C14 C2 179.5(2) . . . . ?
N3 C2 C14 N6 174.7(2) . . . . ?
N2 C2 C14 N6 -4.8(3) . . . . ?
N3 C2 C14 C15 -4.6(3) . . . . ?
N2 C2 C14 C15 175.9(2) . . . . ?
N6 C14 C15 C16 1.3(4) . . . . ?

C2 C14 C15 C16 -179.4(2) . . . . ?
C14 C15 C16 C17 0.0(4) . . . . ?
C15 C16 C17 C18 -1.5(4) . . . . ?
C14 N6 C18 C17 -0.4(4) . . . . ?
C16 C17 C18 N6 1.7(5) . . . . ?
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Cl1' Ni1' N1' C1' -81.37(19) . . . . ?
O1' Ni1' N1' C3' -91.1(2) . . . . ?
N5' Ni1' N1' C3' 179.6(2) . . . . ?
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Cl1' Ni1' N4' C8' 80.7(2) . . . . ?
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O2' Ni1' N4' C4' 174.74(17) . . . . ?
O1' Ni1' N4' C4' 87.86(18) . . . . ?
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Cl1' Ni1' N4' C4' -98.11(17) . . . . ?
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C3' N3' C2' C14' 177.4(2) . . . . ?
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C5' C6' C7' C8' 1.5(4) . . . . ?
C4' N4' C8' C7' -1.2(4) . . . . ?
Ni1' N4' C8' C7' 180.0(2) . . . . ?
C6' C7' C8' N4' -0.3(4) . . . . ?
C13' N5' C9' C10' -0.4(4) . . . . ?
Ni1' N5' C9' C10' -177.0(2) . . . . ?
C13' N5' C9' C1' 178.6(2) . . . . ?
Ni1' N5' C9' C1' 1.9(3) . . . . ?
N1' C1' C9' N5' 0.8(3) . . . . ?
N2' C1' C9' N5' -177.8(2) . . . . ?
N1' C1' C9' C10' 179.8(2) . . . . ?
N2' C1' C9' C10' 1.2(4) . . . . ?
N5' C9' C10' C11' -0.2(4) . . . . ?
C1' C9' C10' C11' -179.1(2) . . . . ?
C9' C10' C11' C12' 0.9(4) . . . . ?
C10' C11' C12' C13' -1.0(4) . . . . ?
C9' N5' C13' C12' 0.2(4) . . . . ?
Ni1' N5' C13' C12' 176.3(2) . . . . ?
C11' C12' C13' N5' 0.4(4) . . . . ?
C18' N6' C14' C15' 1.8(4) . . . . ?
C18' N6' C14' C2' -178.9(2) . . . . ?
N3' C2' C14' N6' -1.5(4) . . . . ?
N2' C2' C14' N6' 177.6(2) . . . . ?
N3' C2' C14' C15' 177.8(2) . . . . ?
N2' C2' C14' C15' -3.1(4) . . . . ?
N6' C14' C15' C16' -0.6(4) . . . . ?
C2' C14' C15' C16' -179.9(2) . . . . ?
C14' C15' C16' C17' -0.9(4) . . . . ?
C15' C16' C17' C18' 1.1(4) . . . . ?
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O1' H1A' Cl10  0.82 2.33 3.143(2) 174.9 .
O1' H1B' Cl1  0.79(3) 2.29(3) 3.063(2) 166(3) .
O2' H2A' Cl1'  0.82 2.38 3.137(2) 153.5 2_676
O2' H2B' Cl10  0.773(17) 2.241(17) 3.012(2) 175(3) .
O10 H10A Cl10  0.737(17) 2.447(18) 3.182(3) 174(4) 1_545
O10 H10B O12  0.733(17) 2.068(19) 2.796(4) 173(4) 1_554
O11 H11A Cl2  0.87(4) 2.31(4) 3.178(3) 174(3) 2_566
O11 H11B Cl1'  0.82(4) 2.35(4) 3.115(3) 155(3) 1_545
O12 H12A Cl10  0.84(4) 2.41(4) 3.213(3) 161(4) 2_666
O12 H12B O11  0.79(4) 2.07(4) 2.837(4) 167(4) .
O13 H13A N6'  0.76(4) 2.16(4) 2.851(3) 151(4) 2_566
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'R.Zibaseresht'

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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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O1 O 0.59251(12) 0.83490(10) 0.74476(12) 0.0208(3) Uani 1 1 d D . .
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H1B H 0.628(2) 0.8806(13) 0.736(2) 0.031 Uiso 1 1 d D . .
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H2B H 0.503(2) 0.8939(14) 1.051(2) 0.042 Uiso 1 1 d D . .
N1 N 0.63618(13) 0.72168(11) 0.93341(12) 0.0156(3) Uani 1 1 d . . .
N2 N 0.80184(14) 0.64066(11) 1.01786(13) 0.0173(3) Uani 1 1 d . . .
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H6X H 0.664(2) 0.4037(17) 0.9481(19) 0.026(6) Uiso 1 1 d . . .
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C6 C 0.27739(17) 0.59373(16) 0.76616(16) 0.0233(5) Uani 1 1 d . . .
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C7 C 0.24086(17) 0.68412(17) 0.74561(17) 0.0243(5) Uani 1 1 d . . .
H7 H 0.1641 0.6969 0.7104 0.029 Uiso 1 1 calc R . .
C8 C 0.31774(17) 0.75574(15) 0.77712(16) 0.0205(4) Uani 1 1 d . . .
H8 H 0.2921 0.8172 0.7621 0.025 Uiso 1 1 calc R . .
C9 C 0.79876(17) 0.81070(14) 1.01158(15) 0.0175(4) Uani 1 1 d . . .
C10 C 0.91095(17) 0.82176(14) 1.06973(16) 0.0191(4) Uani 1 1 d . . .
H10 H 0.9577 0.7701 1.0954 0.036(7) Uiso 1 1 calc R . .
C11 C 0.95237(18) 0.91086(16) 1.08910(16) 0.0244(5) Uani 1 1 d . . .
H11 H 1.0286 0.9212 1.1282 0.026(6) Uiso 1 1 calc R . .
C12 C 0.88136(19) 0.98440(15) 1.05083(17) 0.0265(5) Uani 1 1 d . . .
H12 H 0.9083 1.0457 1.0639 0.022(6) Uiso 1 1 calc R . .
C13 C 0.76961(18) 0.96753(15) 0.99276(17) 0.0232(4) Uani 1 1 d . . .
H13 H 0.7216 1.0182 0.9661 0.028 Uiso 1 1 calc R . .
C14 C 0.79727(17) 0.47489(14) 1.01576(15) 0.0183(4) Uani 1 1 d . . .
C15 C 0.90886(17) 0.46602(15) 1.07395(16) 0.0208(4) Uani 1 1 d . . .
H15 H 0.9531 0.5189 1.1002 0.025 Uiso 1 1 calc R . .
C16 C 0.95579(19) 0.37803(17) 1.09365(18) 0.0276(5) Uani 1 1 d . . .
H16 H 1.0322 0.3707 1.1340 0.033 Uiso 1 1 calc R . .
C17 C 0.8902(2) 0.30149(16) 1.05406(18) 0.0286(5) Uani 1 1 d . . .
H17 H 0.9219 0.2416 1.0655 0.034 Uiso 1 1 calc R . .
C18 C 0.7780(2) 0.31344(15) 0.99759(17) 0.0251(5) Uani 1 1 d . . .
H18 H 0.7320 0.2617 0.9708 0.030 Uiso 1 1 calc R . .
Cl3 Cl 0.98294(5) 0.15738(3) 0.37397(4) 0.02481(12) Uani 1 1 d . . .
Cl4 Cl 0.74869(5) 0.17855(4) 0.77565(5) 0.03120(14) Uani 1 1 d . . .
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H3B H 0.049(2) 0.147(2) 0.228(2) 0.054 Uiso 1 1 d D . .
O4 O 0.77111(15) 0.47025(11) 0.78133(13) 0.0296(4) Uani 1 1 d D . .
H4A H 0.7089(18) 0.4469(18) 0.752(2) 0.044 Uiso 1 1 d D . .
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O2 0.0443(10) 0.0184(8) 0.0228(8) -0.0001(6) 0.0129(7) 0.0033(7)
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C5 0.0189(10) 0.0205(10) 0.0204(10) -0.0012(8) 0.0045(8) -0.0022(8)
C6 0.0155(10) 0.0295(12) 0.0227(11) -0.0031(9) 0.0025(8) -0.0067(8)
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Cl3 0.0295(3) 0.0189(3) 0.0269(3) 0.0008(2) 0.0098(2) 0.0046(2)
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O3 0.0359(10) 0.0428(11) 0.0314(9) -0.0038(8) 0.0137(8) -0.0013(8)
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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H2A H 1.2077 0.4210 0.2972 0.044 Uiso 1 1 d R . .
H2B H 1.1477 0.5146 0.2147 0.044 Uiso 1 1 d R . .
O3 O 0.8714(4) 0.35401(19) 0.45371(18) 0.0383(6) Uani 1 1 d . . .
H3A H 0.9190 0.4032 0.4655 0.057 Uiso 1 1 d R . .
H3B H 0.7582 0.3607 0.4753 0.057 Uiso 1 1 d R . .
N' N 0.3253(8) 0.4014(3) 0.4869(3) 0.0786(15) Uani 1 1 d . . .
O1' O 0.3648(4) 0.3688(2) 0.4134(2) 0.0472(7) Uani 1 1 d . . .
O2' O 0.1830(8) 0.4540(4) 0.4958(4) 0.134(3) Uani 1 1 d . . .
O3' O 0.4543(10) 0.3733(5) 0.5499(4) 0.153(3) Uani 1 1 d . . .
N" N 0.6218(4) 0.1255(3) 0.2071(2) 0.0368(7) Uani 1 1 d . . .
O1" O 0.6986(4) 0.0539(3) 0.2823(2) 0.0541(8) Uani 1 1 d . . .
O2" O 0.5589(4) 0.2149(2) 0.2115(2) 0.0449(6) Uani 1 1 d . . .
O3" O 0.6102(4) 0.1098(2) 0.1256(2) 0.0471(7) Uani 1 1 d . . .

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N2 0.0267(14) 0.0214(12) 0.0213(12) -0.0092(10) -0.0027(10) -0.0041(10)
N3 0.0238(14) 0.0220(13) 0.0284(13) -0.0125(11) -0.0026(10) -0.0017(10)
N4 0.0232(14) 0.0227(13) 0.0260(13) -0.0095(11) -0.0051(10) -0.0005(10)
N5 0.0257(14) 0.0211(12) 0.0225(12) -0.0106(10) -0.0051(10) 0.0006(10)
N6 0.0443(17) 0.0289(14) 0.0233(13) -0.0131(12) -0.0025(12) -0.0075(12)
C1 0.0211(15) 0.0192(14) 0.0215(14) -0.0074(12) -0.0041(11) -0.0030(11)
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C3 0.0189(15) 0.0190(14) 0.0228(14) -0.0098(12) -0.0007(11) -0.0016(11)
C4 0.0205(15) 0.0201(14) 0.0229(15) -0.0079(12) -0.0022(12) -0.0025(11)
C5 0.0249(17) 0.0220(15) 0.0323(17) -0.0108(13) -0.0054(13) 0.0008(12)
C6 0.0258(17) 0.0217(15) 0.0373(18) -0.0053(13) -0.0082(14) 0.0023(12)

C7 0.0262(17) 0.0300(17) 0.0278(17) -0.0032(13) -0.0084(13) -0.0010(13)
C8 0.0278(17) 0.0292(16) 0.0224(15) -0.0068(13) -0.0055(12) -0.0041(13)
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C10 0.0217(16) 0.0235(15) 0.0267(15) -0.0084(13) -0.0066(12) -0.0019(12)
C11 0.0256(17) 0.0235(16) 0.0379(18) -0.0100(14) -0.0104(13) 0.0027(13)
C12 0.0292(18) 0.0240(16) 0.0408(19) -0.0174(14) -0.0054(14) 0.0049(13)
C13 0.0269(17) 0.0259(15) 0.0300(16) -0.0160(13) -0.0030(13) 0.0016(12)
C14 0.0331(18) 0.0246(15) 0.0223(15) -0.0113(13) 0.0021(13) -0.0070(13)
C15 0.059(2) 0.0284(17) 0.0343(19) -0.0179(15) -0.0055(17) 0.0026(16)
C16 0.092(4) 0.040(2) 0.047(2) -0.032(2) 0.000(2) 0.000(2)
C17 0.106(4) 0.045(2) 0.0286(19) -0.0253(18) 0.002(2) -0.010(2)
C18 0.072(3) 0.043(2) 0.0261(18) -0.0198(16) -0.0027(17) -0.0110(19)
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O3' 0.253(7) 0.169(5) 0.061(3) -0.051(3) -0.034(4) -0.091(5)
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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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N1 C3 1.343(4) . ?
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N5 C13 1.353(4) . ?
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N6 C18 1.349(4) . ?
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C1 C9 1.493(4) . ?
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C3 C4 1.497(4) . ?
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O1 H1B 0.8818 . ?
O2 H2A 0.8120 . ?
O2 H2B 0.8959 . ?
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C9 N5 Ni 114.18(18) . . ?
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N2 C2 N3 125.6(3) . . ?
N2 C2 C14 117.8(3) . . ?
N3 C2 C14 116.6(3) . . ?
N3 C3 N1 123.3(3) . . ?
N3 C3 C4 123.2(2) . . ?
N1 C3 C4 113.6(2) . . ?
N4 C4 C5 123.4(3) . . ?
N4 C4 C3 114.0(2) . . ?
C5 C4 C3 122.6(3) . . ?
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C4 C5 H5 121.0 . . ?
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N5 C9 C1 114.0(2) . . ?
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C9 C10 H10 121.0 . . ?
C11 C10 H10 121.0 . . ?
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Ni O1 H1B 113.7 . . ?
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Ni O2 H2A 109.4 . . ?
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Ni O3 H3A 127.2 . . ?
Ni O3 H3B 109.4 . . ?
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O2' N' O3' 126.6(5) . . ?
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O3 Ni N5 C9 -176.20(19) . . . . ?
O2 Ni N5 C9 92.8(2) . . . . ?
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N4 Ni N5 C9 0.4(3) . . . . ?
C2 N2 C1 N1 2.3(4) . . . . ?
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Ni N1 C1 N2 -176.7(2) . . . . ?
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Ni N1 C3 C4 -4.4(3) . . . . ?
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Ni N4 C8 C7 174.6(2) . . . . ?
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_refine_special_details
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
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 'calc w=1/[\s^2^(Fo^2^)+(0.0438P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
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 'Flack H D (1983), Acta Cryst. A39, 876-881'
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N2 N 0.31912(16) -0.1811(2) 0.26881(11) 0.0288(6) Uani 1 1 d . . .
N3 N 0.42420(16) -0.3052(2) 0.32010(11) 0.0296(6) Uani 1 1 d . . .
N4 N 0.49943(16) -0.1277(2) 0.45318(12) 0.0274(6) Uani 1 1 d . . .
N5 N 0.31536(17) 0.0900(2) 0.36294(11) 0.0271(6) Uani 1 1 d . . .
N6 N 0.30232(19) -0.3527(2) 0.17600(13) 0.0405(7) Uani 1 1 d . . .
C1 C 0.33675(19) -0.0977(3) 0.31173(13) 0.0241(7) Uani 1 1 d . . .
C2 C 0.3633(2) -0.2834(3) 0.27457(14) 0.0290(8) Uani 1 1 d . . .
C3 C 0.43606(19) -0.2171(3) 0.36133(13) 0.0247(7) Uani 1 1 d . . .
C4 C 0.49719(18) -0.2271(3) 0.41578(14) 0.0268(7) Uani 1 1 d . . .
C5 C 0.54562(19) -0.3282(3) 0.42764(16) 0.0325(7) Uani 1 1 d . . .
H5 H 0.5415 -0.3945 0.4017 0.039 Uiso 1 1 calc R . .
C6 C 0.6007(2) -0.3273(3) 0.47973(15) 0.0380(9) Uani 1 1 d . . .
H6 H 0.6342 -0.3933 0.4891 0.046 Uiso 1 1 calc R . .
C7 C 0.6048(2) -0.2276(3) 0.51708(17) 0.0393(9) Uani 1 1 d . . .
H7 H 0.6417 -0.2255 0.5516 0.047 Uiso 1 1 calc R . .
C8 C 0.5534(2) -0.1297(3) 0.50296(15) 0.0356(8) Uani 1 1 d . . .
H8 H 0.5565 -0.0633 0.5289 0.043 Uiso 1 1 calc R . .
C9 C 0.2927(2) 0.0187(3) 0.31290(14) 0.0253(8) Uani 1 1 d . . .
C10 C 0.2327(2) 0.0536(3) 0.26748(14) 0.0298(8) Uani 1 1 d . . .
H10 H 0.2180 0.0028 0.2346 0.036 Uiso 1 1 calc R . .
C11 C 0.1950(2) 0.1663(3) 0.27217(16) 0.0376(9) Uani 1 1 d . . .
H11 H 0.1555 0.1921 0.2422 0.045 Uiso 1 1 calc R . .
C12 C 0.2180(2) 0.2387(3) 0.32278(16) 0.0422(9) Uani 1 1 d . . .
H12 H 0.1940 0.3138 0.3270 0.051 Uiso 1 1 calc R . .
C13 C 0.2780(2) 0.1974(3) 0.36736(15) 0.0351(8) Uani 1 1 d . . .
H13 H 0.2924 0.2461 0.4012 0.042 Uiso 1 1 calc R . .
C14 C 0.3426(2) -0.3828(3) 0.23041(15) 0.0319(8) Uani 1 1 d . . .
C15 C 0.3623(2) -0.4995(3) 0.24669(16) 0.0392(9) Uani 1 1 d . . .
H15 H 0.3912 -0.5162 0.2841 0.047 Uiso 1 1 calc R . .
C16 C 0.3380(3) -0.5913(3) 0.20574(18) 0.0462(10) Uani 1 1 d . . .
H16 H 0.3499 -0.6700 0.2155 0.055 Uiso 1 1 calc R . .
C17 C 0.2957(2) -0.5615(3) 0.15025(17) 0.0468(10) Uani 1 1 d . . .
H17 H 0.2785 -0.6199 0.1220 0.056 Uiso 1 1 calc R . .
C18 C 0.2796(2) -0.4423(3) 0.13783(17) 0.0436(9) Uani 1 1 d . . .
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H18 H 0.2511 -0.4235 0.1005 0.052 Uiso 1 1 calc R . .
N1' N 0.41964(16) 0.1384(2) 0.49246(11) 0.0254(6) Uani 1 1 d . . .
N2' N 0.36915(17) 0.2225(2) 0.58816(12) 0.0280(6) Uani 1 1 d . . .
N3' N 0.48812(18) 0.3109(2) 0.53133(11) 0.0301(7) Uani 1 1 d . . .
N4' N 0.51339(16) 0.1236(2) 0.39210(11) 0.0273(6) Uani 1 1 d . . .
N5' N 0.31528(17) -0.0410(2) 0.49559(12) 0.0280(6) Uani 1 1 d . . .
N6' N 0.41032(19) 0.3882(3) 0.68464(13) 0.0411(7) Uani 1 1 d . . .
C1' C 0.3676(2) 0.1385(3) 0.54371(14) 0.0259(7) Uani 1 1 d . . .
C2' C 0.4305(2) 0.3069(3) 0.57927(14) 0.0300(8) Uani 1 1 d . . .
C3' C 0.4780(2) 0.2255(3) 0.48830(13) 0.0249(7) Uani 1 1 d . . .
C4' C 0.53259(19) 0.2179(2) 0.43022(15) 0.0258(6) Uani 1 1 d . . .
C5' C 0.5958(2) 0.3012(3) 0.41559(15) 0.0312(8) Uani 1 1 d . . .
H5' H 0.6070 0.3647 0.4424 0.037 Uiso 1 1 calc R . .
C6' C 0.6418(2) 0.2864(3) 0.35953(15) 0.0334(8) Uani 1 1 d . . .
H6' H 0.6846 0.3401 0.3485 0.040 Uiso 1 1 calc R . .
C7' C 0.6233(2) 0.1917(3) 0.32065(15) 0.0336(8) Uani 1 1 d . . .
H7' H 0.6533 0.1813 0.2831 0.040 Uiso 1 1 calc R . .
C8' C 0.5590(2) 0.1114(3) 0.33831(14) 0.0306(8) Uani 1 1 d . . .
H8' H 0.5474 0.0473 0.3120 0.037 Uiso 1 1 calc R . .
C9' C 0.3090(2) 0.0345(3) 0.54622(14) 0.0256(8) Uani 1 1 d . . .
C10' C 0.2552(2) 0.0118(3) 0.59748(15) 0.0305(8) Uani 1 1 d . . .
H10' H 0.2511 0.0655 0.6307 0.037 Uiso 1 1 calc R . .
C11' C 0.2076(2) -0.0933(3) 0.59798(16) 0.0368(9) Uani 1 1 d . . .
H11' H 0.1729 -0.1122 0.6323 0.044 Uiso 1 1 calc R . .
C12' C 0.2131(2) -0.1688(3) 0.54661(16) 0.0353(8) Uani 1 1 d . . .
H12' H 0.1815 -0.2388 0.5457 0.042 Uiso 1 1 calc R . .
C13' C 0.2663(2) -0.1388(3) 0.49632(15) 0.0336(8) Uani 1 1 d . . .
H13' H 0.2680 -0.1890 0.4615 0.040 Uiso 1 1 calc R . .
C14' C 0.4335(2) 0.4097(3) 0.62430(14) 0.0310(8) Uani 1 1 d . . .
C15' C 0.4570(2) 0.5212(3) 0.60125(17) 0.0426(9) Uani 1 1 d . . .
H15' H 0.4731 0.5307 0.5593 0.051 Uiso 1 1 calc R . .
C16' C 0.4560(3) 0.6178(3) 0.64215(18) 0.0517(11) Uani 1 1 d . . .
H16' H 0.4695 0.6935 0.6279 0.062 Uiso 1 1 calc R . .
C17' C 0.4345(3) 0.5981(4) 0.70439(18) 0.0552(12) Uani 1 1 d . . .
H17' H 0.4344 0.6604 0.7331 0.066 Uiso 1 1 calc R . .
C18' C 0.4129(3) 0.4837(3) 0.72392(17) 0.0528(11) Uani 1 1 d . . .
H18' H 0.3994 0.4720 0.7663 0.063 Uiso 1 1 calc R . .
Cl1 Cl 0.32246(6) 0.49089(7) 0.44204(3) 0.0372(2) Uani 1 1 d . . .
O11 O 0.30039(15) 0.4158(2) 0.49523(11) 0.0481(7) Uani 1 1 d . . .
O12 O 0.3370(2) 0.6103(2) 0.46246(16) 0.0718(9) Uani 1 1 d . . .
O13 O 0.25247(18) 0.4882(2) 0.39627(12) 0.0554(7) Uani 1 1 d . . .
O14 O 0.39959(16) 0.4443(2) 0.41267(12) 0.0518(7) Uani 1 1 d . . .
Cl2 Cl 0.49505(7) 0.01627(8) 0.17630(4) 0.0392(2) Uani 1 1 d . . .
O21 O 0.4905(2) -0.0696(3) 0.22685(13) 0.0677(9) Uani 1 1 d . . .
O22 O 0.57479(16) 0.0807(3) 0.18084(13) 0.0571(8) Uani 1 1 d . . .
O23 O 0.42399(17) 0.0983(3) 0.18060(16) 0.0816(11) Uani 1 1 d . . .
O24 O 0.49058(18) -0.0463(3) 0.11685(11) 0.0585(8) Uani 1 1 d . . .

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N2 0.0270(16) 0.0322(16) 0.0274(14) -0.0052(12) 0.0020(12) 0.0006(12)
N3 0.0322(16) 0.0283(16) 0.0282(14) -0.0051(12) 0.0026(12) 0.0025(12)
N4 0.0285(15) 0.0255(15) 0.0282(13) -0.0008(11) -0.0022(12) 0.0005(12)
N5 0.0308(16) 0.0261(15) 0.0244(14) -0.0007(11) -0.0004(12) 0.0006(12)
N6 0.0483(19) 0.0370(17) 0.0362(17) -0.0101(14) -0.0082(15) -0.0005(15)
C1 0.0257(18) 0.0257(18) 0.0210(16) -0.0017(13) 0.0010(14) -0.0019(13)
C2 0.028(2) 0.034(2) 0.0247(18) -0.0048(15) 0.0021(14) -0.0034(15)
C3 0.0254(18) 0.0261(18) 0.0225(16) -0.0007(13) 0.0037(14) 0.0000(13)
C4 0.0214(17) 0.0288(17) 0.0302(19) -0.0003(14) 0.0022(14) -0.0022(13)
C5 0.0332(18) 0.0261(16) 0.0381(17) -0.0036(17) 0.0020(18) 0.0016(13)
C6 0.029(2) 0.035(2) 0.049(2) 0.0076(17) -0.0062(17) 0.0081(16)
C7 0.035(2) 0.040(2) 0.042(2) 0.0039(17) -0.0174(17) -0.0018(17)
C8 0.039(2) 0.035(2) 0.0331(19) -0.0018(16) -0.0112(16) -0.0021(16)
C9 0.0280(19) 0.0223(18) 0.0257(17) -0.0001(14) 0.0020(14) -0.0057(14)
C10 0.033(2) 0.032(2) 0.0243(17) 0.0008(14) -0.0041(15) -0.0013(16)
C11 0.043(2) 0.032(2) 0.038(2) 0.0099(16) -0.0061(17) 0.0025(16)
C12 0.046(2) 0.033(2) 0.048(2) -0.0023(17) -0.0050(19) 0.0112(17)
C13 0.041(2) 0.032(2) 0.0331(19) -0.0048(15) -0.0030(16) 0.0048(17)
C14 0.033(2) 0.028(2) 0.0342(18) -0.0054(15) 0.0032(16) -0.0039(15)
C15 0.046(2) 0.036(2) 0.0360(19) -0.0078(16) -0.0005(16) -0.0010(17)
C16 0.055(3) 0.026(2) 0.057(3) -0.0125(17) 0.009(2) -0.0064(18)
C17 0.047(2) 0.046(2) 0.047(2) -0.0218(19) 0.0033(19) -0.0054(19)
C18 0.043(2) 0.046(2) 0.041(2) -0.0131(18) -0.0058(18) -0.0015(18)
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N2' 0.0335(16) 0.0252(15) 0.0254(14) -0.0049(12) -0.0025(12) 0.0003(13)
N3' 0.0352(18) 0.0266(16) 0.0286(15) -0.0031(12) -0.0026(12) 0.0008(12)
N4' 0.0288(16) 0.0252(15) 0.0279(15) -0.0037(12) -0.0003(12) -0.0009(12)
N5' 0.0311(16) 0.0258(15) 0.0271(15) -0.0019(11) 0.0002(12) -0.0036(12)
N6' 0.047(2) 0.0418(18) 0.0348(16) -0.0113(13) 0.0006(14) -0.0052(15)
C1' 0.0288(19) 0.0243(18) 0.0246(17) 0.0023(14) -0.0023(14) 0.0054(14)
C2' 0.034(2) 0.0279(18) 0.0277(18) 0.0001(14) -0.0030(15) 0.0018(15)
C3' 0.0264(18) 0.0242(17) 0.0242(16) 0.0007(13) -0.0042(13) 0.0031(14)
C4' 0.0274(17) 0.0243(15) 0.0256(16) -0.0039(15) -0.0012(16) 0.0024(12)
C5' 0.0316(18) 0.0286(17) 0.033(2) -0.0052(14) -0.0050(15) -0.0042(15)
C6' 0.031(2) 0.035(2) 0.0346(19) 0.0021(16) 0.0046(16) -0.0043(15)
C7' 0.0286(19) 0.041(2) 0.0313(18) -0.0022(16) 0.0034(15) 0.0025(16)
C8' 0.033(2) 0.0302(19) 0.0282(17) -0.0049(14) 0.0012(15) 0.0034(15)
C9' 0.028(2) 0.0243(18) 0.0243(16) 0.0012(14) 0.0018(14) 0.0033(15)
C10' 0.038(2) 0.0282(19) 0.0258(17) -0.0028(14) 0.0004(14) 0.0022(16)
C11' 0.035(2) 0.043(2) 0.0322(18) 0.0053(16) 0.0049(15) 0.0012(17)
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C12' 0.038(2) 0.0286(19) 0.0390(19) 0.0037(16) 0.0002(16) -0.0075(16)
C13' 0.033(2) 0.033(2) 0.0346(19) -0.0048(16) -0.0025(16) -0.0008(16)
C14' 0.033(2) 0.033(2) 0.0269(16) -0.0079(14) 0.0000(14) -0.0032(15)
C15' 0.053(3) 0.035(2) 0.040(2) -0.0081(17) 0.0072(17) -0.0063(18)
C16' 0.062(3) 0.034(2) 0.058(3) -0.0136(19) 0.010(2) -0.010(2)
C17' 0.066(3) 0.050(3) 0.050(2) -0.027(2) 0.012(2) -0.010(2)
C18' 0.062(3) 0.060(3) 0.037(2) -0.0183(19) 0.007(2) -0.009(2)
Cl1 0.0423(5) 0.0284(5) 0.0408(6) -0.0004(4) -0.0002(4) 0.0027(4)
O11 0.0388(16) 0.0604(18) 0.0450(15) 0.0132(13) -0.0025(12) -0.0062(13)
O12 0.084(2) 0.0344(17) 0.097(2) -0.0245(16) 0.0131(18) -0.0089(15)
O13 0.0540(18) 0.0564(18) 0.0559(16) 0.0090(14) -0.0175(13) 0.0111(13)
O14 0.0461(16) 0.0517(15) 0.0576(18) 0.0057(13) 0.0081(13) 0.0157(13)
Cl2 0.0318(4) 0.0540(6) 0.0319(4) -0.0046(5) 0.0027(3) -0.0014(4)
O21 0.092(2) 0.071(2) 0.0397(16) 0.0105(15) 0.0039(15) -0.0374(18)
O22 0.0390(17) 0.066(2) 0.0658(18) 0.0146(16) 0.0015(14) -0.0156(13)
O23 0.0431(19) 0.088(2) 0.113(3) -0.049(2) -0.0031(18) 0.0195(16)
O24 0.0637(19) 0.079(2) 0.0331(14) -0.0125(13) -0.0003(13) 0.0194(16)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Ni N1' 2.054(2) . ?
Ni N4' 2.211(3) . ?
Ni N5' 2.216(3) . ?
Ni N5 2.220(3) . ?
Ni N4 2.225(3) . ?
N1 C3 1.373(4) . ?
N1 C1 1.385(4) . ?
N2 C1 1.365(4) . ?
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N3 C2 1.403(4) . ?
N4 C8 1.377(4) . ?
N4 C4 1.403(4) . ?
N5 C13 1.375(4) . ?

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N5 C9 1.406(4) . ?
N6 C18 1.370(4) . ?
N6 C14 1.384(4) . ?
C1 C9 1.513(4) . ?
C2 C14 1.527(4) . ?
C3 C4 1.531(4) . ?
C4 C5 1.419(4) . ?
C5 C6 1.427(4) . ?
C5 H5 0.9500 . ?
C6 C7 1.405(5) . ?
C6 H6 0.9500 . ?
C7 C8 1.426(5) . ?
C7 H7 0.9500 . ?
C8 H8 0.9500 . ?
C9 C10 1.426(5) . ?
C10 C11 1.433(5) . ?
C10 H10 0.9500 . ?
C11 C12 1.425(5) . ?
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C12 C13 1.437(5) . ?
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C5' H5' 0.9500 . ?
C6' C7' 1.408(4) . ?
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C6' C5' H5' 121.0 . . ?
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C7' C6' H6' 120.2 . . ?

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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
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C1 C 0.3140(3) 0.0568(2) 0.23035(17) 0.0239(5) Uani 1 1 d . . .
C2 C 0.3539(3) 0.1159(2) 0.35069(18) 0.0265(6) Uani 1 1 d . . .
C3 C 0.2507(3) -0.0392(2) 0.37931(18) 0.0260(6) Uani 1 1 d . . .
C4 C 0.1861(4) -0.1366(2) 0.43970(18) 0.0276(6) Uani 1 1 d . . .
C5 C 0.1642(4) -0.1574(2) 0.53364(19) 0.0348(6) Uani 1 1 d . . .
H5 H 0.1904 -0.1085 0.5630 0.042 Uiso 1 1 calc R . .
C6 C 0.1031(4) -0.2514(3) 0.5843(2) 0.0432(8) Uani 1 1 d . . .
H6 H 0.0859 -0.2692 0.6500 0.052 Uiso 1 1 calc R . .
C7 C 0.0672(5) -0.3190(3) 0.5392(2) 0.0471(8) Uani 1 1 d . . .
H7 H 0.0263 -0.3847 0.5735 0.057 Uiso 1 1 calc R . .
C8 C 0.0900(4) -0.2923(2) 0.4446(2) 0.0411(7) Uani 1 1 d . . .
H8 H 0.0632 -0.3401 0.4141 0.049 Uiso 1 1 calc R . .
C9 C 0.3017(3) 0.0585(2) 0.13363(18) 0.0252(5) Uani 1 1 d . . .
C10 C 0.3287(4) 0.1415(2) 0.05974(19) 0.0326(6) Uani 1 1 d . . .
H10 H 0.3627 0.2035 0.0675 0.039 Uiso 1 1 calc R . .
C11 C 0.3055(4) 0.1333(3) -0.0268(2) 0.0411(7) Uani 1 1 d . . .
H11 H 0.3236 0.1901 -0.0797 0.049 Uiso 1 1 calc R . .
C12 C 0.2570(4) 0.0443(3) -0.0361(2) 0.0427(8) Uani 1 1 d . . .
H12 H 0.2427 0.0375 -0.0956 0.051 Uiso 1 1 calc R . .
C13 C 0.2287(4) -0.0357(2) 0.0410(2) 0.0366(7) Uani 1 1 d . . .
H13 H 0.1928 -0.0980 0.0343 0.044 Uiso 1 1 calc R . .
C14 C 0.3994(4) 0.1982(2) 0.38631(18) 0.0291(6) Uani 1 1 d . . .
C15 C 0.3659(4) 0.1899(2) 0.4786(2) 0.0365(7) Uani 1 1 d . . .
H15 H 0.3189 0.1291 0.5215 0.044 Uiso 1 1 calc R . .
C16 C 0.4013(5) 0.2704(3) 0.5080(2) 0.0442(8) Uani 1 1 d . . .
H16 H 0.3801 0.2663 0.5719 0.053 Uiso 1 1 calc R . .
C17 C 0.4671(5) 0.3565(3) 0.4451(2) 0.0455(8) Uani 1 1 d . . .
H17 H 0.4893 0.4141 0.4636 0.055 Uiso 1 1 calc R . .
C18 C 0.5006(4) 0.3582(2) 0.3545(2) 0.0397(7) Uani 1 1 d . . .
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H18 H 0.5494 0.4179 0.3106 0.048 Uiso 1 1 calc R . .
N1 N 0.2575(3) -0.02894(16) 0.29004(14) 0.0239(5) Uani 1 1 d . . .
N2 N 0.3713(3) 0.13223(16) 0.25828(14) 0.0247(5) Uani 1 1 d . . .
N3 N 0.2956(3) 0.03240(17) 0.41301(15) 0.0299(5) Uani 1 1 d . . .
N4 N 0.1482(3) -0.20231(17) 0.39442(16) 0.0300(5) Uani 1 1 d . . .
N5 N 0.2496(3) -0.02975(17) 0.12483(15) 0.0285(5) Uani 1 1 d . . .
N6 N 0.4684(3) 0.28068(17) 0.32490(16) 0.0312(5) Uani 1 1 d . . .
N7 N -0.1885(3) -0.06109(18) 0.29261(16) 0.0318(5) Uani 1 1 d . . .
O71 O -0.0909(3) -0.10393(15) 0.24270(14) 0.0351(5) Uani 1 1 d . . .
O72 O -0.1241(3) -0.03028(17) 0.35023(14) 0.0410(5) Uani 1 1 d . . .
O73 O -0.3517(3) -0.05202(18) 0.28107(17) 0.0516(6) Uani 1 1 d . . .
N8 N 0.1808(3) 0.3466(2) 0.15220(19) 0.0415(6) Uani 1 1 d . . .
O81 O 0.3469(3) 0.34083(16) 0.12165(14) 0.0370(5) Uani 1 1 d . . .
O82 O 0.0675(3) 0.3644(2) 0.09791(19) 0.0694(8) Uani 1 1 d . . .
O83 O 0.1402(4) 0.3343(4) 0.2319(2) 0.1102(15) Uani 1 1 d . . .
N9 N 0.7944(3) 0.1716(2) 0.1536(2) 0.0410(6) Uani 1 1 d . . .
O91 O 0.7669(3) 0.18581(16) 0.23179(15) 0.0408(5) Uani 1 1 d . . .
O92 O 0.6788(3) 0.21006(18) 0.09844(15) 0.0442(5) Uani 1 1 d . . .
O93 O 0.9196(3) 0.1244(2) 0.1331(2) 0.0661(8) Uani 1 1 d . . .
N10 N 0.3063(4) 0.3887(2) 0.6746(2) 0.0502(7) Uani 1 1 d . . .
O101 O 0.2471(4) 0.3086(2) 0.7247(2) 0.0837(10) Uani 1 1 d . . .
O102 O 0.2045(5) 0.4515(3) 0.6416(3) 0.1086(13) Uani 1 1 d . . .
O103 O 0.4734(4) 0.3986(2) 0.6588(2) 0.0683(8) Uani 1 1 d . . .
O1 O 0.4417(3) -0.1920(2) 0.23914(19) 0.0449(6) Uani 1 1 d . . .
H1A H 0.5151 -0.1497 0.2458 0.067 Uiso 1 1 calc R . .
H1B H 0.463(6) -0.241(3) 0.263(3) 0.055(15) Uiso 1 1 d . . .
O2 O 0.0925(3) -0.25498(18) 0.20833(18) 0.0501(6) Uani 1 1 d . . .
H2 H -0.009(5) -0.275(3) 0.230(2) 0.042(10) Uiso 1 1 d . . .
C21 C 0.1854(6) -0.3228(4) 0.1686(4) 0.0834(15) Uani 1 1 d . . .
H21A H 0.2922 -0.2870 0.1277 0.100 Uiso 1 1 calc R . .
H21B H 0.2327 -0.3757 0.2192 0.100 Uiso 1 1 calc R . .
C22 C 0.0919(9) -0.3679(6) 0.1186(6) 0.158(4) Uani 1 1 d . . .
H22A H -0.0249 -0.3945 0.1538 0.237 Uiso 1 1 calc R . .
H22B H 0.1632 -0.4225 0.1061 0.237 Uiso 1 1 calc R . .
H22C H 0.0697 -0.3192 0.0599 0.237 Uiso 1 1 calc R . .
O3 O 0.7075(3) 0.38902(16) 0.15834(16) 0.0398(5) Uani 1 1 d . . .
H3 H 0.804(6) 0.376(3) 0.153(3) 0.063(14) Uiso 1 1 d . . .
C31 C 0.6879(5) 0.4764(3) 0.0838(2) 0.0505(9) Uani 1 1 d . . .
H31A H 0.6209 0.4588 0.0371 0.061 Uiso 1 1 calc R . .
H31B H 0.8101 0.5027 0.0536 0.061 Uiso 1 1 calc R . .
C32 C 0.5913(8) 0.5535(4) 0.1139(3) 0.0880(16) Uani 1 1 d . . .
H32A H 0.4768 0.5255 0.1508 0.132 Uiso 1 1 calc R . .
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H32C H 0.6669 0.5794 0.1517 0.132 Uiso 1 1 calc R . .

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C1 0.0162(12) 0.0327(15) 0.0232(13) -0.0089(11) -0.0014(9) 0.0016(10)
C2 0.0256(14) 0.0306(15) 0.0232(13) -0.0084(11) -0.0007(10) -0.0012(10)
C3 0.0214(13) 0.0336(15) 0.0224(13) -0.0074(11) -0.0028(10) 0.0002(10)
C4 0.0257(13) 0.0312(15) 0.0245(14) -0.0059(11) -0.0030(10) 0.0000(10)
C5 0.0339(16) 0.0425(18) 0.0259(15) -0.0068(13) -0.0034(11) -0.0013(12)
C6 0.0430(18) 0.051(2) 0.0278(16) 0.0008(14) -0.0034(13) -0.0032(14)
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C8 0.0448(18) 0.0319(17) 0.0455(19) -0.0068(14) -0.0132(14) -0.0028(13)
C9 0.0196(12) 0.0359(15) 0.0220(13) -0.0112(11) -0.0019(9) 0.0007(10)
C10 0.0326(15) 0.0389(17) 0.0263(15) -0.0091(13) -0.0043(11) -0.0017(12)
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C12 0.0466(19) 0.062(2) 0.0216(15) -0.0153(15) -0.0057(12) -0.0027(15)
C13 0.0396(17) 0.0465(19) 0.0289(16) -0.0180(14) -0.0071(12) -0.0004(13)
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C17 0.059(2) 0.0422(19) 0.0431(19) -0.0251(16) -0.0012(15) -0.0049(15)
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O72 0.0356(12) 0.0590(15) 0.0342(12) -0.0217(11) -0.0067(9) 0.0045(9)
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O82 0.0337(14) 0.101(2) 0.0636(17) -0.0061(16) -0.0133(12) 0.0060(13)
O83 0.0498(19) 0.222(4) 0.0428(17) -0.020(2) 0.0086(13) 0.036(2)
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O92 0.0338(12) 0.0634(15) 0.0385(12) -0.0210(11) -0.0005(9) 0.0021(10)
O93 0.0406(15) 0.0629(17) 0.098(2) -0.0353(16) 0.0104(13) 0.0139(12)
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O101 0.0588(18) 0.0422(17) 0.125(3) 0.0053(17) 0.0194(17) 0.0030(13)
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O103 0.0559(18) 0.0677(19) 0.083(2) -0.0277(16) 0.0023(14) -0.0142(13)
O1 0.0317(12) 0.0505(17) 0.0560(16) -0.0197(14) -0.0088(10) 0.0103(11)
O2 0.0395(14) 0.0513(15) 0.0729(18) -0.0411(13) 0.0000(12) -0.0027(11)
C21 0.063(3) 0.084(3) 0.134(4) -0.082(3) -0.007(3) 0.008(2)
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C22 0.124(6) 0.202(8) 0.214(8) -0.176(7) 0.025(5) -0.023(5)
O3 0.0310(13) 0.0355(13) 0.0465(13) -0.0031(10) 0.0000(10) -0.0022(9)
C31 0.054(2) 0.044(2) 0.047(2) -0.0034(16) -0.0036(16) -0.0035(15)
C32 0.128(5) 0.073(3) 0.069(3) -0.029(3) -0.015(3) 0.036(3)

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 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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C1 C9 1.472(3) . ?
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C3 N3 1.310(3) . ?
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C3 C4 1.455(4) . ?
C4 N4 1.346(3) . ?
C4 C5 1.361(4) . ?
C5 C6 1.370(4) . ?
C5 H5 0.9500 . ?
C6 C7 1.362(5) . ?
C6 H6 0.9500 . ?
C7 C8 1.365(5) . ?

C7 H7 0.9500 . ?
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C8 H8 0.9500 . ?
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C9 C10 1.358(4) . ?
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C16 C17 1.357(5) . ?
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_diffrn_reflns_limit_l_min        -17
_diffrn_reflns_limit_l_max        17
_diffrn_reflns_theta_min          1.97
_diffrn_reflns_theta_max          26.40
_reflns_number_total              4720
_reflns_number_gt                 3805
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        'Bruker SMART'
_computing_cell_refinement        'Bruker SAINT+'
_computing_data_reduction         'Bruker XPREP'
_computing_structure_solution     'SHELXS-97 (Sheldrick, 1990)'
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_computing_molecular_graphics     'Bruker SHELXTL'
_computing_publication_material   'Bruker SHELXTL'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0303P)^2^+3.6056P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
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_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     'constr'
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          4720
_refine_ls_number_parameters      334
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0435
_refine_ls_R_factor_gt            0.0274
_refine_ls_wR_factor_ref          0.0720
_refine_ls_wR_factor_gt           0.0650
_refine_ls_goodness_of_fit_ref    1.046
_refine_ls_restrained_S_all       1.046
_refine_ls_shift/su_max           0.001
_refine_ls_shift/su_mean          0.000

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N1 N 0.9277(2) 0.32075(16) 0.64312(16) 0.0149(5) Uani 1 1 d . . .
N2 N 1.0093(2) 0.25414(16) 0.63340(16) 0.0161(5) Uani 1 1 d . . .
N3 N 0.7595(2) 0.18394(16) 0.60146(16) 0.0151(5) Uani 1 1 d . . .
N4 N 0.8663(2) 0.13771(16) 0.59743(16) 0.0164(5) Uani 1 1 d . . .
N5 N 0.8546(2) 0.27799(16) 0.45507(16) 0.0151(5) Uani 1 1 d . . .
N6 N 0.9457(2) 0.21583(16) 0.47401(16) 0.0161(5) Uani 1 1 d . . .
N7 N 0.7491(2) 0.44202(16) 0.51121(16) 0.0151(5) Uani 1 1 d . . .
N8 N 0.5925(2) 0.31274(16) 0.47296(16) 0.0157(5) Uani 1 1 d . . .
Cl Cl 0.65659(6) 0.35673(4) 0.69453(4) 0.01221(14) Uani 1 1 d . . .
C1 C 1.1142(3) 0.2625(2) 0.69530(19) 0.0192(6) Uani 1 1 d . . .
H1 H 1.1836 0.2234 0.7023 0.023 Uiso 1 1 calc R . .
C2 C 1.1011(3) 0.3375(2) 0.7453(2) 0.0209(6) Uani 1 1 d . . .
H2 H 1.1596 0.3619 0.7936 0.025 Uiso 1 1 calc R . .
C3 C 0.9841(3) 0.3715(2) 0.7111(2) 0.0187(6) Uani 1 1 d . . .
H3 H 0.9496 0.4240 0.7337 0.022 Uiso 1 1 calc R . .
C4 C 0.8577(3) 0.05472(19) 0.6316(2) 0.0211(6) Uani 1 1 d . . .
H4 H 0.9211 0.0108 0.6372 0.025 Uiso 1 1 calc R . .
```

C5 C 0.7415(3) 0.0458(2) 0.6562(2) 0.0223(7) Uani 1 1 d . . .
H5 H 0.7070 -0.0055 0.6814 0.027 Uiso 1 1 calc R . .
C6 C 0.6832(3) 0.1273(2) 0.63700(19) 0.0188(6) Uani 1 1 d . . .
H6 H 0.6004 0.1407 0.6477 0.023 Uiso 1 1 calc R . .
C7 C 1.0058(3) 0.2005(2) 0.3981(2) 0.0195(6) Uani 1 1 d . . .
H7 H 1.0729 0.1605 0.3947 0.023 Uiso 1 1 calc R . .
C8 C 0.9511(3) 0.2537(2) 0.3279(2) 0.0214(6) Uani 1 1 d . . .
H8 H 0.9717 0.2578 0.2655 0.026 Uiso 1 1 calc R . .
C9 C 0.8588(3) 0.3007(2) 0.3660(2) 0.0190(6) Uani 1 1 d . . .
H9 H 0.8059 0.3434 0.3327 0.023 Uiso 1 1 calc R . .
C10 C 0.9722(3) 0.1823(2) 0.56928(19) 0.0172(6) Uani 1 1 d . . .
H10 H 1.0426 0.1390 0.5717 0.021 Uiso 1 1 calc R . .
C11 C 0.8359(3) 0.5052(2) 0.5317(2) 0.0204(6) Uani 1 1 d . . .
H11 H 0.9094 0.4905 0.5723 0.024 Uiso 1 1 calc R . .
C12 C 0.8237(3) 0.5897(2) 0.4971(2) 0.0246(7) Uani 1 1 d . . .
H12 H 0.8874 0.6323 0.5135 0.029 Uiso 1 1 calc R . .
C13 C 0.7180(3) 0.6121(2) 0.4381(2) 0.0254(7) Uani 1 1 d . . .
H13 H 0.7066 0.6707 0.4143 0.031 Uiso 1 1 calc R . .
C14 C 0.6291(3) 0.5483(2) 0.4142(2) 0.0219(6) Uani 1 1 d . . .
H14 H 0.5562 0.5621 0.3725 0.026 Uiso 1 1 calc R . .
C15 C 0.6462(3) 0.46369(19) 0.4511(2) 0.0171(6) Uani 1 1 d . . .
C16 C 0.5586(3) 0.3904(2) 0.42942(19) 0.0174(6) Uani 1 1 d . . .
C17 C 0.4492(3) 0.3975(2) 0.3680(2) 0.0224(7) Uani 1 1 d . . .
H17 H 0.4266 0.4523 0.3377 0.027 Uiso 1 1 calc R . .
C18 C 0.3735(3) 0.3242(2) 0.3513(2) 0.0234(7) Uani 1 1 d . . .
H18 H 0.2982 0.3279 0.3093 0.028 Uiso 1 1 calc R . .
C19 C 0.4080(3) 0.2457(2) 0.3959(2) 0.0226(7) Uani 1 1 d . . .
H19 H 0.3568 0.1946 0.3855 0.027 Uiso 1 1 calc R . .
C20 C 0.5184(3) 0.2423(2) 0.4562(2) 0.0188(6) Uani 1 1 d . . .
H20 H 0.5423 0.1879 0.4868 0.023 Uiso 1 1 calc R . .
P P 0.31286(8) 0.06020(6) 0.60222(6) 0.02344(18) Uani 1 1 d . . .
F1 F 0.40902(17) 0.05834(13) 0.52834(14) 0.0351(5) Uani 1 1 d . . .
F2 F 0.34530(18) 0.16270(12) 0.62394(13) 0.0308(4) Uani 1 1 d . . .
F3 F 0.20571(17) 0.09089(13) 0.52082(13) 0.0301(4) Uani 1 1 d . . .
F4 F 0.21193(19) 0.06444(13) 0.67578(13) 0.0355(5) Uani 1 1 d . . .
F5 F 0.27808(18) -0.04036(12) 0.58085(13) 0.0325(5) Uani 1 1 d . . .
F6 F 0.4176(2) 0.03297(14) 0.68453(15) 0.0425(6) Uani 1 1 d . . .

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N2 0.0164(11) 0.0185(12) 0.0131(11) -0.0003(10) 0.0007(10) 0.0033(10)

N3 0.0146(11) 0.0186(12) 0.0115(11) -0.0001(10) -0.0001(9) 0.0028(10)
N4 0.0185(12) 0.0145(12) 0.0156(12) -0.0003(10) 0.0000(10) 0.0025(10)
N5 0.0134(11) 0.0177(12) 0.0136(11) 0.0009(10) -0.0006(9) 0.0022(10)
N6 0.0178(11) 0.0181(12) 0.0121(11) -0.0012(9) 0.0005(9) 0.0039(10)
N7 0.0162(11) 0.0172(12) 0.0122(11) 0.0009(9) 0.0030(9) 0.0024(10)
N8 0.0149(11) 0.0209(12) 0.0114(11) 0.0003(10) 0.0022(9) 0.0012(10)
Cl 0.0127(3) 0.0140(3) 0.0104(3) -0.0001(2) 0.0030(2) 0.0012(2)
C1 0.0150(13) 0.0277(17) 0.0142(14) 0.0021(12) -0.0010(11) 0.0006(12)
C2 0.0198(14) 0.0271(17) 0.0152(14) -0.0008(12) 0.0000(12) -0.0010(12)
C3 0.0207(14) 0.0206(16) 0.0150(14) -0.0020(12) 0.0030(12) -0.0031(12)
C4 0.0318(16) 0.0138(14) 0.0161(14) -0.0012(12) -0.0028(13) 0.0010(13)
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C6 0.0213(14) 0.0223(16) 0.0122(13) -0.0007(12) -0.0003(12) -0.0051(12)
C7 0.0182(13) 0.0245(16) 0.0163(14) -0.0052(12) 0.0040(11) 0.0031(12)
C8 0.0215(15) 0.0291(17) 0.0131(14) -0.0014(12) 0.0010(12) 0.0017(13)
C9 0.0188(14) 0.0238(16) 0.0138(13) 0.0014(12) 0.0000(11) 0.0008(12)
C10 0.0192(13) 0.0184(14) 0.0134(13) 0.0005(12) -0.0007(11) 0.0022(12)
C11 0.0182(14) 0.0222(16) 0.0215(15) 0.0020(12) 0.0052(12) 0.0000(12)
C12 0.0265(16) 0.0175(15) 0.0314(17) 0.0034(13) 0.0099(14) -0.0020(13)
C13 0.0302(17) 0.0210(16) 0.0275(16) 0.0076(13) 0.0132(14) 0.0053(13)
C14 0.0234(15) 0.0238(16) 0.0196(15) 0.0057(13) 0.0072(13) 0.0079(13)
C15 0.0183(14) 0.0207(15) 0.0136(13) 0.0014(11) 0.0072(11) 0.0042(12)
C16 0.0196(14) 0.0217(15) 0.0117(13) 0.0001(11) 0.0046(11) 0.0051(12)
C17 0.0221(15) 0.0318(18) 0.0129(14) 0.0042(13) 0.0012(12) 0.0084(14)
C18 0.0190(14) 0.0362(19) 0.0141(14) -0.0013(13) -0.0014(12) 0.0037(14)
C19 0.0196(14) 0.0321(18) 0.0159(14) -0.0035(13) 0.0012(12) -0.0028(13)
C20 0.0189(14) 0.0237(16) 0.0138(13) -0.0007(12) 0.0021(11) 0.0003(12)
P 0.0245(4) 0.0220(4) 0.0224(4) -0.0005(3) -0.0025(3) 0.0037(3)
F1 0.0278(10) 0.0362(12) 0.0424(12) -0.0099(9) 0.0083(9) 0.0026(9)
F2 0.0378(11) 0.0229(10) 0.0319(10) -0.0025(8) 0.0047(9) 0.0015(8)
F3 0.0282(10) 0.0370(11) 0.0239(9) 0.0023(8) -0.0009(8) 0.0131(9)
F4 0.0453(12) 0.0359(12) 0.0267(10) 0.0011(9) 0.0104(9) -0.0038(10)
F5 0.0428(11) 0.0233(10) 0.0285(10) -0.0020(8) -0.0060(9) 0.0013(9)
F6 0.0468(13) 0.0325(12) 0.0415(12) 0.0009(10) -0.0203(10) 0.0026(10)

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;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Ru N7 2.035(2) . ?
Ru N8 2.044(2) . ?
Ru N1 2.063(2) . ?
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Ru Cl 2.4114(7) . ?
N1 C3 1.325(4) . ?
N1 N2 1.357(3) . ?
N2 C1 1.354(3) . ?
N2 C10 1.444(4) . ?
N3 C6 1.334(4) . ?
N3 N4 1.357(3) . ?
N4 C4 1.351(4) . ?
N4 C10 1.430(4) . ?
N5 C9 1.326(4) . ?
N5 N6 1.363(3) . ?
N6 C7 1.355(4) . ?
N6 C10 1.449(3) . ?
N7 C11 1.344(4) . ?
N7 C15 1.359(4) . ?
N8 C20 1.334(4) . ?
N8 C16 1.356(4) . ?
C1 C2 1.355(4) . ?
C1 H1 0.9500 . ?
C2 C3 1.395(4) . ?
C2 H2 0.9500 . ?
C3 H3 0.9500 . ?
C4 C5 1.355(4) . ?
C4 H4 0.9500 . ?
C5 C6 1.393(4) . ?
C5 H5 0.9500 . ?
C6 H6 0.9500 . ?
C7 C8 1.362(4) . ?
C7 H7 0.9500 . ?
C8 C9 1.392(4) . ?
C8 H8 0.9500 . ?
C9 H9 0.9500 . ?
C10 H10 1.0000 . ?
C11 C12 1.367(4) . ?
C11 H11 0.9500 . ?
C12 C13 1.375(4) . ?
C12 H12 0.9500 . ?
C13 C14 1.373(5) . ?
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C14 C15 1.384(4) . ?
C14 H14 0.9500 . ?
C15 C16 1.463(4) . ?
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C16 C17 1.387(4) . ?
C17 C18 1.379(5) . ?
C17 H17 0.9500 . ?
C18 C19 1.375(4) . ?
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C19 C20 1.383(4) . ?
C19 H19 0.9500 . ?
C20 H20 0.9500 . ?
P F1 1.576(2) . ?
P F6 1.582(2) . ?
P F5 1.583(2) . ?
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N5 Ru N8 92.12(9) . . ?
N7 Ru N8 79.05(9) . . ?
N5 Ru N1 85.88(9) . . ?
N7 Ru N1 99.01(9) . . ?
N8 Ru N1 177.22(9) . . ?
N5 Ru N3 87.64(9) . . ?
N7 Ru N3 176.24(9) . . ?
N8 Ru N3 97.74(9) . . ?
N1 Ru N3 84.12(9) . . ?
N5 Ru Cl 175.26(6) . . ?
N7 Ru Cl 91.49(7) . . ?
N8 Ru Cl 92.48(7) . . ?
N1 Ru Cl 89.56(7) . . ?
N3 Ru Cl 90.62(7) . . ?
C3 N1 N2 104.9(2) . . ?
C3 N1 Ru 137.8(2) . . ?
N2 N1 Ru 117.17(17) . . ?
C1 N2 N1 111.5(2) . . ?
C1 N2 C10 128.9(2) . . ?
N1 N2 C10 119.3(2) . . ?
C6 N3 N4 105.0(2) . . ?
C6 N3 Ru 137.6(2) . . ?
N4 N3 Ru 117.32(17) . . ?
C4 N4 N3 111.2(2) . . ?
C4 N4 C10 129.2(2) . . ?
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N3 N4 C10 119.2(2) . . ?
C9 N5 N6 104.8(2) . . ?
C9 N5 Ru 136.8(2) . . ?
N6 N5 Ru 118.35(17) . . ?
C7 N6 N5 111.5(2) . . ?
C7 N6 C10 129.6(2) . . ?
N5 N6 C10 118.6(2) . . ?
C11 N7 C15 117.5(2) . . ?
C11 N7 Ru 126.35(19) . . ?
C15 N7 Ru 116.16(19) . . ?
C20 N8 C16 119.0(2) . . ?
C20 N8 Ru 125.6(2) . . ?
C16 N8 Ru 115.42(19) . . ?
N2 C1 C2 106.7(3) . . ?
N2 C1 H1 126.6 . . ?
C2 C1 H1 126.6 . . ?
C1 C2 C3 105.8(3) . . ?
C1 C2 H2 127.1 . . ?
C3 C2 H2 127.1 . . ?
N1 C3 C2 111.0(3) . . ?
N1 C3 H3 124.5 . . ?
C2 C3 H3 124.5 . . ?
N4 C4 C5 107.2(3) . . ?
N4 C4 H4 126.4 . . ?
C5 C4 H4 126.4 . . ?
C4 C5 C6 105.8(3) . . ?
C4 C5 H5 127.1 . . ?
C6 C5 H5 127.1 . . ?
N3 C6 C5 110.7(3) . . ?
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C5 C6 H6 124.7 . . ?
N6 C7 C8 106.5(3) . . ?
N6 C7 H7 126.8 . . ?
C8 C7 H7 126.8 . . ?
C7 C8 C9 106.0(3) . . ?
C7 C8 H8 127.0 . . ?
C9 C8 H8 127.0 . . ?
N5 C9 C8 111.1(3) . . ?
N5 C9 H9 124.4 . . ?
C8 C9 H9 124.4 . . ?
N4 C10 N2 110.0(2) . . ?
N4 C10 N6 111.1(2) . . ?
N2 C10 N6 110.0(2) . . ?
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N2 C10 H10 108.6 . . ?
N6 C10 H10 108.6 . . ?
N7 C11 C12 123.3(3) . . ?
N7 C11 H11 118.3 . . ?
C12 C11 H11 118.3 . . ?

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C11 C12 C13 119.0(3) . . ?
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N7 C15 C14 121.5(3) . . ?
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N8 C16 C15 115.2(2) . . ?
C17 C16 C15 123.7(3) . . ?
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C16 C17 H17 120.4 . . ?
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C18 C19 C20 118.9(3) . . ?
C18 C19 H19 120.5 . . ?
C20 C19 H19 120.5 . . ?
N8 C20 C19 122.3(3) . . ?
N8 C20 H20 118.9 . . ?
C19 C20 H20 118.9 . . ?
F1 P F6 91.34(12) . . ?
F1 P F5 90.82(11) . . ?
F6 P F5 91.45(11) . . ?
F1 P F3 89.77(11) . . ?
F6 P F3 177.97(12) . . ?
F5 P F3 90.23(11) . . ?
F1 P F2 90.01(11) . . ?
F6 P F2 89.28(11) . . ?
F5 P F2 178.87(12) . . ?
F3 P F2 89.02(11) . . ?
F1 P F4 178.17(12) . . ?
F6 P F4 90.27(12) . . ?
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N7 Ru N1 C3 51.8(3) . . . . ?
N3 Ru N1 C3 -130.3(3) . . . . ?
Cl Ru N1 C3 -39.6(3) . . . . ?
N5 Ru N1 N2 -42.31(19) . . . . ?
N7 Ru N1 N2 -132.16(19) . . . . ?
N3 Ru N1 N2 45.74(19) . . . . ?
Cl Ru N1 N2 136.41(18) . . . . ?
C3 N1 N2 C1 0.9(3) . . . . ?
Ru N1 N2 C1 -176.36(18) . . . . ?
C3 N1 N2 C10 175.0(2) . . . . ?
Ru N1 N2 C10 -2.2(3) . . . . ?
N5 Ru N3 C6 -143.6(3) . . . . ?
N8 Ru N3 C6 -51.8(3) . . . . ?
N1 Ru N3 C6 130.3(3) . . . . ?
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Ru N3 N4 C4 176.31(18) . . . . ?
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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C17 C 0.5130(4) 0.6179(3) -0.0274(4) 0.0342(11) Uani 1 1 d . . .
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C18 C 0.4687(4) 0.6163(3) -0.1356(4) 0.0376(12) Uani 1 1 d . . .
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C19 C 0.3658(4) 0.6151(3) -0.1902(4) 0.0349(11) Uani 1 1 d . . .
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C20 C 0.3097(4) 0.6175(3) -0.1367(3) 0.0292(10) Uani 1 1 d . . .
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H1B H 0.2905 0.4723 0.1158 0.061 Uiso 1 1 d R . .
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O12 O 0.2642(3) 0.2986(3) 0.2453(4) 0.0849(17) Uani 1 1 d . . .
O13 O 0.3803(4) 0.4116(5) 0.2667(4) 0.111(2) Uani 1 1 d . . .
O14 O 0.2800(3) 0.4220(3) 0.3507(4) 0.0777(15) Uani 1 1 d . . .
Cl2 Cl 0.10067(8) 0.45388(7) 0.65245(8) 0.0253(2) Uani 1 1 d . . .
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O24 O 0.1155(3) 0.5505(2) 0.6446(3) 0.0406(8) Uani 1 1 d . . .
C99 C 0.2003(6) 0.2251(5) 0.4682(6) 0.091(2) Uani 1 1 d . . .
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H99B H 0.1337 0.2421 0.4182 0.136 Uiso 1 1 calc R . .
H99C H 0.2469 0.2705 0.4681 0.136 Uiso 1 1 calc R . .
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C5 0.026(2) 0.042(3) 0.032(2) -0.004(2) 0.009(2) -0.002(2)
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C12 0.045(3) 0.035(3) 0.028(2) 0.004(2) 0.002(2) 0.007(2)
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C19 0.044(3) 0.033(3) 0.037(3) -0.005(2) 0.027(2) -0.002(2)
C20 0.032(3) 0.028(2) 0.031(2) -0.0030(18) 0.017(2) 0.0028(19)
O1 0.056(3) 0.0267(18) 0.0312(19) 0.0030(14) 0.0125(19) -0.0022(16)
Cl1 0.0420(7) 0.0400(7) 0.0326(6) 0.0055(5) 0.0149(5) 0.0079(6)
O11 0.040(2) 0.057(2) 0.040(2) 0.0129(18) 0.0115(18) 0.0090(18)
O12 0.044(3) 0.080(3) 0.094(4) -0.051(3) -0.001(3) 0.003(2)
O13 0.053(3) 0.182(6) 0.088(4) 0.077(4) 0.023(3) -0.010(3)
O14 0.064(3) 0.065(3) 0.083(3) -0.022(2) 0.015(3) 0.025(2)
Cl2 0.0262(6) 0.0242(5) 0.0263(5) -0.0004(4) 0.0126(5) -0.0048(4)
O21 0.038(2) 0.059(3) 0.045(2) 0.0137(19) 0.0062(18) 0.0095(19)
O22 0.0337(19) 0.049(2) 0.048(2) -0.0131(17) 0.0234(17) -0.0167(16)
O23 0.059(2) 0.040(2) 0.047(2) -0.0171(16) 0.038(2) -0.0184(18)
O24 0.058(2) 0.0261(18) 0.044(2) -0.0003(15) 0.0283(19) -0.0096(16)
C99 0.113(7) 0.071(5) 0.080(5) -0.006(4) 0.037(5) 0.011(5)
O99 0.233(9) 0.073(4) 0.066(4) 0.001(3) 0.053(5) 0.012(4)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
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 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Ru O1 2.151(3) . ?
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N1 N2 1.362(5) . ?
N2 C1 1.354(5) . ?
N2 C10 1.454(5) . ?
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C1 N2 C10 127.7(4) . . ?
N1 N2 C10 119.1(3) . . ?
C6 N3 N4 104.7(3) . . ?
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C4 N4 N3 111.1(3) . . ?
C4 N4 C10 129.0(3) . . ?
N3 N4 C10 118.6(3) . . ?
C9 N5 N6 105.3(3) . . ?
C9 N5 Ru 135.5(3) . . ?
N6 N5 Ru 119.1(3) . . ?
C7 N6 N5 111.0(3) . . ?
C7 N6 C10 130.3(3) . . ?
N5 N6 C10 118.6(3) . . ?
C11 N7 C15 118.4(4) . . ?
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C15 N7 Ru 115.6(3) . . ?
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N8 C16 C15 114.3(4) . . ?
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O14 Cl1 O12 107.5(3) . . ?
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O13 Cl1 O12 108.6(4) . . ?
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_reflns_number_gt                3790
_reflns_threshold_expression     >2sigma(I)

_computing_data_collection       'Bruker SMART'
_computing_cell_refinement       'Bruker SAINT+'
_computing_data_reduction        'Bruker XPREP'
_computing_structure_solution    'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement  'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics    'Bruker SHELXTL'
_computing_publication_material  'Bruker SHELXTL'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type           full
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0339P)^2^+0.6211P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    ,constr,
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       ?
_refine_ls_number_reflns         5207
_refine_ls_number_parameters     481
_refine_ls_number_restraints     72
_refine_ls_R_factor_all          0.0651
_refine_ls_R_factor_gt           0.0355
_refine_ls_wR_factor_ref         0.0753
_refine_ls_wR_factor_gt          0.0677
_refine_ls_goodness_of_fit_ref   1.011
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Ru Ru 0.75399(2) 1.046720(14) 0.69993(2) 0.01335(9) Uani 1 1 d . . .
N1 N 0.88271(19) 0.99939(13) 0.6560(2) 0.0121(6) Uani 1 1 d . . .
N2 N 0.6920(2) 0.96194(15) 0.6291(2) 0.0174(6) Uani 1 1 d . A .
N3 N 0.6002(2) 1.06381(16) 0.7112(2) 0.0217(7) Uani 1 1 d . . .
N4 N 0.7942(2) 1.00393(14) 0.8533(2) 0.0160(6) Uani 1 1 d . . .
N5 N 0.8131(2) 1.13294(14) 0.7933(2) 0.0177(7) Uani 1 1 d . . .
C1 C 0.9796(2) 1.02466(18) 0.6693(3) 0.0157(8) Uani 1 1 d . . .
H1 H 0.9929 1.0699 0.7005 0.019 Uiso 1 1 calc R . .
C2 C 1.0611(2) 0.98744(18) 0.6392(3) 0.0174(8) Uani 1 1 d . . .
H2 H 1.1292 1.0066 0.6502 0.021 Uiso 1 1 calc R . .
C3 C 1.0417(3) 0.92176(18) 0.5927(3) 0.0177(8) Uani 1 1 d . . .
H3 H 1.0964 0.8952 0.5715 0.021 Uiso 1 1 calc R . .
C4 C 0.9418(3) 0.89539(18) 0.5775(3) 0.0171(8) Uani 1 1 d . . .
H4 H 0.9274 0.8504 0.5459 0.021 Uiso 1 1 calc R . .
C5 C 0.8628(2) 0.93475(16) 0.6085(3) 0.0136(7) Uani 1 1 d . . .
C6 C 0.7531(2) 0.91239(18) 0.5933(3) 0.0164(8) Uani 1 1 d . . .
C7 C 0.7107(3) 0.84960(19) 0.5521(3) 0.0222(8) Uani 1 1 d . A .
H7 H 0.7537 0.8152 0.5272 0.027 Uiso 1 1 calc R . .
C8 C 0.6048(3) 0.8365(2) 0.5470(3) 0.0308(10) Uani 1 1 d . . .
C9 C 0.5438(3) 0.8891(2) 0.5829(3) 0.0322(10) Uani 1 1 d . A .
H9 H 0.4714 0.8818 0.5795 0.039 Uiso 1 1 calc R . .
C10 C 0.5884(3) 0.9513(2) 0.6232(3) 0.0228(8) Uani 1 1 d . . .
C11 C 0.5352(3) 1.0110(2) 0.6650(3) 0.0277(9) Uani 1 1 d . A .
C12 C 0.4292(3) 1.0159(3) 0.6588(3) 0.0445(13) Uani 1 1 d . . .
H12 H 0.3854 0.9792 0.6262 0.053 Uiso 1 1 calc R A .
C13 C 0.3867(3) 1.0739(3) 0.6999(4) 0.0598(16) Uani 1 1 d . A .
H13 H 0.3137 1.0774 0.6962 0.072 Uiso 1 1 calc R . .
C14 C 0.4512(3) 1.1268(3) 0.7463(4) 0.0526(15) Uani 1 1 d . . .
H14 H 0.4235 1.1672 0.7757 0.063 Uiso 1 1 calc R A .
C15 C 0.5568(3) 1.1203(2) 0.7495(3) 0.0338(10) Uani 1 1 d . A .
H15 H 0.6008 1.1574 0.7802 0.041 Uiso 1 1 calc R . .
C16 C 0.5672(9) 0.7573(6) 0.5225(10) 0.018(3) Uani 0.484(4) 1 d PU A 1
C17 C 0.6312(5) 0.6978(4) 0.5271(6) 0.0195(18) Uani 0.484(4) 1 d PU A 1
H17 H 0.7045 0.7028 0.5457 0.023 Uiso 0.484(4) 1 calc PR A 1
C18 C 0.5888(6) 0.6327(4) 0.5048(6) 0.0208(18) Uani 0.484(4) 1 d PU A 1
H18 H 0.6334 0.5932 0.5103 0.025 Uiso 0.484(4) 1 calc PR A 1
C19 C 0.4825(5) 0.6230(4) 0.4745(6) 0.0190(18) Uani 0.484(4) 1 d PU A 1
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C20 C 0.4188(12) 0.6811(8) 0.4639(14) 0.017(4) Uani 0.484(4) 1 d PU A 1
H20 H 0.3462 0.6757 0.4395 0.021 Uiso 0.484(4) 1 calc PR A 1
C21 C 0.4607(13) 0.7481(9) 0.4889(10) 0.018(3) Uani 0.484(4) 1 d PU A 1
H21 H 0.4158 0.7875 0.4829 0.021 Uiso 0.484(4) 1 calc PR A 1
C22 C 0.4341(6) 0.5508(4) 0.4591(7) 0.034(2) Uani 0.484(4) 1 d P A 1
H22A H 0.4140 0.5355 0.5286 0.051 Uiso 0.484(4) 1 calc PR A 1
H22B H 0.4847 0.5179 0.4380 0.051 Uiso 0.484(4) 1 calc PR A 1
H22C H 0.3726 0.5524 0.4007 0.051 Uiso 0.484(4) 1 calc PR A 1
C16' C 0.5455(8) 0.7788(5) 0.4929(9) 0.016(2) Uani 0.516(4) 1 d PU A 2
C17' C 0.5841(5) 0.7398(3) 0.4127(6) 0.0196(17) Uani 0.516(4) 1 d PU A 2
H17' H 0.6475 0.7528 0.3903 0.024 Uiso 0.516(4) 1 calc PR A 2
C18' C 0.5291(5) 0.6821(4) 0.3663(6) 0.0247(18) Uani 0.516(4) 1 d PU A 2
H18' H 0.5556 0.6564 0.3110 0.030 Uiso 0.516(4) 1 calc PR A 2
C19' C 0.4377(6) 0.6601(4) 0.3967(7) 0.0239(19) Uani 0.516(4) 1 d PU A 2
C20' C 0.4017(12) 0.6989(7) 0.4808(14) 0.017(3) Uani 0.516(4) 1 d PU A 2
H20' H 0.3405 0.6845 0.5063 0.020 Uiso 0.516(4) 1 calc PR A 2
C21' C 0.4541(12) 0.7573(8) 0.5260(10) 0.015(3) Uani 0.516(4) 1 d PU A 2
H21' H 0.4274 0.7833 0.5808 0.018 Uiso 0.516(4) 1 calc PR A 2
C22' C 0.3769(6) 0.5977(4) 0.3433(7) 0.040(2) Uani 0.516(4) 1 d P A 2
H22D H 0.3171 0.5892 0.3799 0.060 Uiso 0.516(4) 1 calc PR A 2
H22E H 0.4218 0.5563 0.3512 0.060 Uiso 0.516(4) 1 calc PR A 2
H22F H 0.3525 0.6072 0.2645 0.060 Uiso 0.516(4) 1 calc PR A 2
C27 C 0.8401(2) 1.04723(19) 0.9358(3) 0.0179(7) Uani 1 1 d . . .
C26 C 0.8763(3) 1.0216(2) 1.0415(3) 0.0260(9) Uani 1 1 d . . .
H26 H 0.9064 1.0526 1.0988 0.031 Uiso 1 1 calc R . .
C25 C 0.8685(4) 0.9515(2) 1.0632(3) 0.0438(12) Uani 1 1 d . . .
H25 H 0.8929 0.9336 1.1353 0.053 Uiso 1 1 calc R . .
C24 C 0.8251(4) 0.9079(2) 0.9791(4) 0.0501(14) Uani 1 1 d . . .
H24 H 0.8203 0.8592 0.9920 0.060 Uiso 1 1 calc R . .
C23 C 0.7886(3) 0.93500(19) 0.8762(3) 0.0298(10) Uani 1 1 d . . .
H23 H 0.7581 0.9042 0.8188 0.036 Uiso 1 1 calc R . .
C28 C 0.8480(2) 1.12024(18) 0.9032(3) 0.0172(8) Uani 1 1 d . . .
C29 C 0.8835(3) 1.17383(19) 0.9765(3) 0.0242(9) Uani 1 1 d . . .
H29 H 0.9096 1.1639 1.0521 0.029 Uiso 1 1 calc R . .
C30 C 0.8806(3) 1.2418(2) 0.9380(3) 0.0352(11) Uani 1 1 d . . .
H30 H 0.9030 1.2793 0.9872 0.042 Uiso 1 1 calc R . .
C31 C 0.8449(4) 1.2544(2) 0.8278(4) 0.0427(12) Uani 1 1 d . . .
H31 H 0.8428 1.3008 0.7999 0.051 Uiso 1 1 calc R . .
C32 C 0.8120(3) 1.19943(19) 0.7577(3) 0.0295(9) Uani 1 1 d . . .
H32 H 0.7877 1.2089 0.6815 0.035 Uiso 1 1 calc R . .
Cl Cl 0.73042(6) 1.10768(5) 0.52525(7) 0.0197(2) Uani 1 1 d . . .
P P 0.87683(8) 0.72956(5) 0.79178(9) 0.0269(2) Uani 1 1 d . . .
F1 F 0.87036(19) 0.74273(11) 0.92010(18) 0.0386(6) Uani 1 1 d . . .
F2 F 0.92689(17) 0.80506(11) 0.78612(18) 0.0336(5) Uani 1 1 d . . .
F3 F 0.76364(18) 0.76173(15) 0.7585(2) 0.0552(7) Uani 1 1 d . . .
F4 F 0.99039(19) 0.69730(12) 0.82850(19) 0.0421(6) Uani 1 1 d . . .
F5 F 0.8296(2) 0.65265(13) 0.8007(2) 0.0610(8) Uani 1 1 d . . .
F6 F 0.88361(18) 0.71626(12) 0.66518(18) 0.0405(6) Uani 1 1 d . . .

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N1 0.0145(14) 0.0095(16) 0.0113(16) -0.0004(12) -0.0005(10) 0.0016(11)
N2 0.0146(14) 0.0238(18) 0.0137(16) -0.0048(13) 0.0023(11) -0.0012(12)
N3 0.0125(15) 0.037(2) 0.0155(17) -0.0060(14) 0.0006(12) 0.0056(12)
N4 0.0150(15) 0.0170(17) 0.0153(17) -0.0045(13) 0.0011(11) -0.0021(12)
N5 0.0243(17) 0.0130(17) 0.0164(18) 0.0002(13) 0.0054(12) 0.0007(12)
C1 0.0170(18) 0.016(2) 0.0122(19) -0.0014(15) -0.0023(13) -0.0033(13)
C2 0.0118(17) 0.020(2) 0.019(2) 0.0037(17) -0.0002(13) -0.0016(14)
C3 0.0184(19) 0.019(2) 0.017(2) 0.0055(16) 0.0062(14) 0.0048(14)
C4 0.0212(19) 0.0119(19) 0.019(2) 0.0000(15) 0.0067(14) 0.0017(14)
C5 0.0165(18) 0.013(2) 0.0108(19) 0.0023(14) 0.0006(13) -0.0002(12)
C6 0.0167(18) 0.020(2) 0.0121(19) -0.0026(16) 0.0023(13) -0.0031(14)
C7 0.025(2) 0.021(2) 0.022(2) -0.0090(17) 0.0101(15) -0.0074(15)
C8 0.030(2) 0.046(3) 0.019(2) -0.0182(19) 0.0118(17) -0.0224(18)
C9 0.018(2) 0.058(3) 0.023(2) -0.022(2) 0.0093(16) -0.0190(18)
C10 0.0165(18) 0.038(2) 0.0145(19) -0.0071(19) 0.0048(13) -0.0023(17)
C11 0.0145(19) 0.052(3) 0.016(2) -0.0140(19) 0.0019(14) -0.0033(17)
C12 0.017(2) 0.087(4) 0.028(3) -0.031(2) 0.0003(16) -0.002(2)
C13 0.017(2) 0.122(5) 0.039(3) -0.049(3) -0.0006(19) 0.013(2)
C14 0.024(2) 0.087(4) 0.042(3) -0.045(3) -0.0066(19) 0.026(2)
C15 0.027(2) 0.049(3) 0.023(2) -0.016(2) -0.0021(16) 0.0120(18)
C16 0.020(5) 0.018(6) 0.015(6) 0.000(4) -0.001(4) -0.002(4)
C17 0.015(4) 0.019(4) 0.025(4) -0.006(3) 0.007(3) -0.003(3)
C18 0.029(4) 0.011(4) 0.022(4) -0.007(3) 0.006(3) 0.001(3)
C19 0.022(4) 0.020(4) 0.016(4) -0.001(3) 0.007(3) -0.005(3)
C20 0.013(6) 0.015(7) 0.022(7) -0.006(5) -0.002(5) -0.009(5)
C21 0.023(5) 0.014(6) 0.015(7) 0.001(5) 0.001(5) 0.007(4)
C22 0.038(5) 0.019(5) 0.045(6) -0.012(4) 0.011(4) -0.013(4)
C16' 0.016(5) 0.015(5) 0.016(5) 0.001(4) 0.001(4) -0.001(4)
C17' 0.013(3) 0.017(4) 0.029(4) -0.006(3) 0.003(3) 0.000(3)
C18' 0.024(4) 0.022(4) 0.028(4) -0.011(3) 0.004(3) 0.000(3)
C19' 0.026(4) 0.020(4) 0.026(5) -0.010(4) 0.005(3) -0.004(3)
C20' 0.018(5) 0.007(6) 0.026(6) -0.005(4) 0.008(4) 0.000(4)
C21' 0.023(5) 0.010(5) 0.012(6) -0.004(5) -0.001(5) 0.001(3)
C22' 0.037(5) 0.031(5) 0.055(6) -0.023(4) 0.020(4) -0.013(4)
C27 0.0194(17) 0.0177(19) 0.0161(19) -0.0026(17) 0.0015(13) 0.0033(15)
C26 0.033(2) 0.027(2) 0.015(2) -0.0033(17) -0.0038(16) 0.0039(16)
C25 0.082(4) 0.025(3) 0.019(2) 0.003(2) -0.006(2) 0.014(2)
C24 0.112(4) 0.014(2) 0.023(3) 0.003(2) 0.007(2) -0.002(2)
C23 0.055(3) 0.016(2) 0.018(2) -0.0025(17) 0.0053(18) -0.0103(17)
C28 0.0131(18) 0.019(2) 0.020(2) -0.0052(16) 0.0022(14) -0.0022(13)
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C29 0.025(2) 0.027(2) 0.021(2) -0.0040(18) 0.0041(15) -0.0048(16)
C30 0.059(3) 0.023(2) 0.027(3) -0.015(2) 0.017(2) -0.0150(19)
C31 0.084(4) 0.012(2) 0.038(3) -0.005(2) 0.027(2) -0.010(2)
C32 0.053(3) 0.013(2) 0.025(2) 0.0023(18) 0.0117(18) 0.0038(17)
Cl 0.0160(4) 0.0252(5) 0.0174(5) 0.0040(4) 0.0018(3) 0.0052(3)
P 0.0395(6) 0.0167(6) 0.0290(6) 0.0006(5) 0.0186(5) -0.0016(4)
F1 0.0711(18) 0.0222(13) 0.0308(14) 0.0027(11) 0.0321(12) 0.0010(11)
F2 0.0524(15) 0.0177(12) 0.0340(14) 0.0054(10) 0.0172(11) -0.0033(10)
F3 0.0363(15) 0.079(2) 0.0537(17) -0.0014(15) 0.0178(12) 0.0085(13)
F4 0.0582(16) 0.0308(14) 0.0410(15) 0.0059(12) 0.0190(12) 0.0205(11)
F5 0.097(2) 0.0301(15) 0.068(2) -0.0128(14) 0.0480(17) -0.0310(14)
F6 0.0469(15) 0.0469(16) 0.0316(14) -0.0121(12) 0.0173(11) -0.0100(11)

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;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Ru N4 2.033(3) . ?
Ru N1 2.064(3) . ?
Ru N3 2.065(3) . ?
Ru N5 2.079(3) . ?
Ru Cl 2.4104(9) . ?
N1 C1 1.340(4) . ?
N1 C5 1.373(4) . ?
N2 C10 1.359(4) . ?
N2 C6 1.361(4) . ?
N3 C15 1.343(4) . ?
N3 C11 1.378(5) . ?
N4 C23 1.354(4) . ?
N4 C27 1.364(4) . ?
N5 C32 1.345(4) . ?
N5 C28 1.366(4) . ?
C1 C2 1.384(5) . ?
C1 H1 0.9500 . ?
C2 C3 1.385(5) . ?
C2 H2 0.9500 . ?

C3 C4 1.383(5) . ?
C3 H3 0.9500 . ?
C4 C5 1.383(4) . ?
C4 H4 0.9500 . ?
C5 C6 1.478(4) . ?
C6 C7 1.382(5) . ?
C7 C8 1.398(5) . ?
C7 H7 0.9500 . ?
C8 C9 1.402(5) . ?
C8 C16' 1.443(11) . ?
C8 C16 1.606(12) . ?
C9 C10 1.379(5) . ?
C9 H9 0.9500 . ?
C10 C11 1.475(5) . ?
C11 C12 1.379(5) . ?
C12 C13 1.375(6) . ?
C12 H12 0.9500 . ?
C13 C14 1.376(6) . ?
C13 H13 0.9500 . ?
C14 C15 1.381(5) . ?
C14 H14 0.9500 . ?
C15 H15 0.9500 . ?
C16 C21 1.39(2) . ?
C16 C17 1.409(13) . ?
C17 C18 1.372(10) . ?
C17 H17 0.9500 . ?
C18 C19 1.387(10) . ?
C18 H18 0.9500 . ?
C19 C20 1.381(16) . ?
C19 C22 1.518(10) . ?
C20 C21 1.41(2) . ?
C20 H20 0.9500 . ?
C21 H21 0.9500 . ?
C22 H22A 0.9800 . ?
C22 H22B 0.9800 . ?
C22 H22C 0.9800 . ?
C16' C21' 1.390(19) . ?
C16' C17' 1.396(13) . ?
C17' C18' 1.386(9) . ?
C17' H17' 0.9500 . ?
C18' C19' 1.378(10) . ?
C18' H18' 0.9500 . ?
C19' C20' 1.416(14) . ?
C19' C22' 1.518(10) . ?
C20' C21' 1.37(2) . ?
C20' H20' 0.9500 . ?
C21' H21' 0.9500 . ?
C22' H22D 0.9800 . ?
C22' H22E 0.9800 . ?

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C22' H22F 0.9800 . ?
C27 C26 1.388(5) . ?
C27 C28 1.462(5) . ?
C26 C25 1.376(5) . ?
C26 H26 0.9500 . ?
C25 C24 1.370(6) . ?
C25 H25 0.9500 . ?
C24 C23 1.370(5) . ?
C24 H24 0.9500 . ?
C23 H23 0.9500 . ?
C28 C29 1.389(5) . ?
C29 C30 1.382(5) . ?
C29 H29 0.9500 . ?
C30 C31 1.371(6) . ?
C30 H30 0.9500 . ?
C31 C32 1.379(5) . ?
C31 H31 0.9500 . ?
C32 H32 0.9500 . ?
P F3 1.589(3) . ?
P F6 1.590(2) . ?
P F2 1.593(2) . ?
P F4 1.599(2) . ?
P F5 1.607(2) . ?
P F1 1.610(2) . ?

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N2 Ru N1 79.24(10) . . ?
N4 Ru N1 88.46(10) . . ?
N2 Ru N3 80.06(11) . . ?
N4 Ru N3 95.71(11) . . ?
N1 Ru N3 159.18(11) . . ?
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N2 N 0.7536(5) 0.7754(2) 0.7525(3) 0.0208(10) Uani 1 1 d . A .
N3 N 0.9064(5) 0.6871(2) 0.7354(3) 0.0244(11) Uani 1 1 d . A .
N4 N 0.7567(5) 0.7279(3) 0.6068(3) 0.0248(11) Uani 1 1 d . A .
N5 N 0.6769(5) 0.6193(2) 0.6399(3) 0.0245(11) Uani 1 1 d . A .
C1 C 0.4323(6) 0.7201(3) 0.6434(4) 0.0299(15) Uani 1 1 d . . .
H1 H 0.4347 0.6833 0.6170 0.036 Uiso 1 1 calc R A .
C2 C 0.3190(7) 0.7506(3) 0.6401(4) 0.0361(16) Uani 1 1 d . A .
H2 H 0.2454 0.7354 0.6112 0.043 Uiso 1 1 calc R . .
C3 C 0.3134(7) 0.8038(3) 0.6795(4) 0.0363(16) Uani 1 1 d . . .
H3 H 0.2354 0.8245 0.6796 0.044 Uiso 1 1 calc R A .
C4 C 0.4238(6) 0.8261(3) 0.7186(4) 0.0300(14) Uani 1 1 d . A .
H4 H 0.4225 0.8628 0.7455 0.036 Uiso 1 1 calc R . .
C5 C 0.5356(6) 0.7949(3) 0.7182(3) 0.0236(13) Uani 1 1 d . . .
C6 C 0.6597(6) 0.8162(3) 0.7564(3) 0.0217(13) Uani 1 1 d . A .
C7 C 0.6854(6) 0.8721(3) 0.7901(3) 0.0230(13) Uani 1 1 d . . .
H7 H 0.6193 0.9010 0.7917 0.028 Uiso 1 1 calc R A .
C8 C 0.8093(6) 0.8866(3) 0.8222(3) 0.0217(12) Uani 1 1 d . A .
C9 C 0.9030(6) 0.8419(3) 0.8191(3) 0.0221(13) Uani 1 1 d . . .
H9 H 0.9871 0.8494 0.8415 0.027 Uiso 1 1 calc R A .
C10 C 0.8739(6) 0.7872(3) 0.7836(3) 0.0211(12) Uani 1 1 d . A .
C11 C 0.9618(6) 0.7367(3) 0.7726(3) 0.0244(13) Uani 1 1 d . . .
C12 C 1.0900(7) 0.7380(3) 0.7970(4) 0.0316(15) Uani 1 1 d . A .
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C13 C 1.1644(7) 0.6883(4) 0.7837(4) 0.0404(18) Uani 1 1 d . . .
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C14 C 1.1086(7) 0.6387(4) 0.7461(4) 0.0410(19) Uani 1 1 d . A .
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C15 C 0.9793(7) 0.6396(3) 0.7229(4) 0.0321(15) Uani 1 1 d . . .
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C16 C 0.8401(6) 0.9469(3) 0.8572(3) 0.0232(13) Uani 1 1 d . . .
C17 C 0.7452(6) 0.9878(3) 0.8698(3) 0.0247(13) Uani 1 1 d . A .
H17 H 0.6592 0.9767 0.8557 0.030 Uiso 1 1 calc R . .
C18 C 0.7743(6) 1.0443(3) 0.9023(3) 0.0254(13) Uani 1 1 d . . .
H18 H 0.7079 1.0712 0.9097 0.030 Uiso 1 1 calc R A .
C19 C 0.8987(6) 1.0623(3) 0.9242(3) 0.0275(14) Uani 1 1 d . A .
C20 C 0.9925(7) 1.0222(3) 0.9105(4) 0.0346(16) Uani 1 1 d . . .
H20 H 1.0784 1.0336 0.9243 0.042 Uiso 1 1 calc R A .
C21 C 0.9648(6) 0.9661(3) 0.8773(4) 0.0314(15) Uani 1 1 d . A .
H21 H 1.0316 0.9402 0.8680 0.038 Uiso 1 1 calc R . .
C22 C 0.9312(7) 1.1233(3) 0.9598(4) 0.0355(16) Uani 1 1 d . . .
H22A H 0.9583 1.1525 0.9264 0.053 Uiso 1 1 calc R A .
H22B H 0.9996 1.1175 0.9994 0.053 Uiso 1 1 calc R . .
H22C H 0.8567 1.1399 0.9767 0.053 Uiso 1 1 calc R . .
C23 C 0.8033(7) 0.7840(3) 0.5946(3) 0.0302(15) Uani 1 1 d . . .
```

H23 H 0.8139 0.8135 0.6313 0.036 Uiso 1 1 calc R A .
C24 C 0.8363(7) 0.8003(3) 0.5312(4) 0.0368(16) Uani 1 1 d . A .
H24 H 0.8715 0.8399 0.5250 0.044 Uiso 1 1 calc R . .
C25 C 0.8174(8) 0.7582(4) 0.4769(4) 0.0392(18) Uani 1 1 d . . .
H25 H 0.8369 0.7688 0.4323 0.047 Uiso 1 1 calc R A .
C26 C 0.7700(7) 0.7009(3) 0.4881(3) 0.0328(15) Uani 1 1 d . A .
H26 H 0.7567 0.6715 0.4511 0.039 Uiso 1 1 calc R . .
C27 C 0.7414(6) 0.6856(3) 0.5537(3) 0.0260(14) Uani 1 1 d . . .
C28 C 0.6969(6) 0.6251(3) 0.5724(3) 0.0258(14) Uani 1 1 d . A .
C29 C 0.6785(6) 0.5752(3) 0.5265(4) 0.0310(15) Uani 1 1 d . . .
H29 H 0.6911 0.5799 0.4793 0.037 Uiso 1 1 calc R A .
C30 C 0.6419(6) 0.5189(3) 0.5498(4) 0.0337(16) Uani 1 1 d . A .
H30 H 0.6319 0.4841 0.5193 0.040 Uiso 1 1 calc R . .
C31 C 0.6199(6) 0.5134(3) 0.6177(4) 0.0332(16) Uani 1 1 d . . .
H31 H 0.5933 0.4752 0.6346 0.040 Uiso 1 1 calc R A .
C32 C 0.6374(6) 0.5650(3) 0.6609(4) 0.0298(15) Uani 1 1 d . A .
H32 H 0.6206 0.5615 0.7074 0.036 Uiso 1 1 calc R . .
N6 N 0.681(5) 0.656(3) 0.794(3) 0.020(6) Uani 0.66(2) 1 d PU A 2
C33 C 0.590(3) 0.673(2) 0.830(2) 0.027(6) Uani 0.66(2) 1 d PU A 2
H33 H 0.5343 0.7053 0.8102 0.032 Uiso 0.66(2) 1 calc PR A 2
C34 C 0.570(2) 0.6482(12) 0.8923(12) 0.026(4) Uani 0.66(2) 1 d PU A 2
H34 H 0.5004 0.6614 0.9131 0.031 Uiso 0.66(2) 1 calc PR A 2
C35 C 0.6518(18) 0.6037(8) 0.9249(10) 0.023(4) Uani 0.66(2) 1 d PU A 2
C36 C 0.7504(18) 0.5868(9) 0.8906(10) 0.023(4) Uani 0.66(2) 1 d PU A 2
H36 H 0.8101 0.5570 0.9113 0.028 Uiso 0.66(2) 1 calc PR A 2
C37 C 0.763(3) 0.6124(14) 0.8273(17) 0.025(5) Uani 0.66(2) 1 d PU A 2
H37 H 0.8313 0.5994 0.8053 0.031 Uiso 0.66(2) 1 calc PR A 2
C38 C 0.6415(15) 0.5749(6) 0.9921(6) 0.029(3) Uani 0.66(2) 1 d PU A 2
H38 H 0.7093 0.5493 1.0128 0.035 Uiso 0.66(2) 1 calc PR A 2
C39 C 0.5460(12) 0.5814(5) 1.0261(7) 0.032(3) Uani 0.66(2) 1 d PU A 2
H39 H 0.4782 0.6074 1.0062 0.039 Uiso 0.66(2) 1 calc PR A 2
C40 C 0.5377(18) 0.5508(7) 1.0935(6) 0.027(3) Uani 0.66(2) 1 d PU A 2
C41 C 0.6353(15) 0.5181(9) 1.1319(8) 0.032(3) Uani 0.66(2) 1 d PU A 2
H41 H 0.7133 0.5136 1.1154 0.039 Uiso 0.66(2) 1 calc PR A 2
C42 C 0.618(2) 0.4918(9) 1.1949(10) 0.036(4) Uani 0.66(2) 1 d PU A 2
H42 H 0.6869 0.4698 1.2209 0.044 Uiso 0.66(2) 1 calc PR A 2
N7 N 0.512(5) 0.4954(18) 1.221(2) 0.027(5) Uani 0.66(2) 1 d PU A 2
C43 C 0.416(3) 0.526(2) 1.1830(19) 0.027(5) Uani 0.66(2) 1 d PU A 2
H43 H 0.3389 0.5291 1.2004 0.033 Uiso 0.66(2) 1 calc PR A 2
C44 C 0.4241(18) 0.5540(8) 1.1189(8) 0.028(3) Uani 0.66(2) 1 d PU A 2
H44 H 0.3533 0.5747 1.0932 0.034 Uiso 0.66(2) 1 calc PRU A 2
N6' N 0.667(11) 0.663(6) 0.798(6) 0.026(12) Uani 0.34(2) 1 d PU A 1
C33' C 0.735(6) 0.619(3) 0.832(4) 0.028(9) Uani 0.34(2) 1 d PU A 1
H33' H 0.8052 0.6035 0.8130 0.034 Uiso 0.34(2) 1 calc PR A 1
C34' C 0.709(4) 0.594(2) 0.895(2) 0.027(7) Uani 0.34(2) 1 d PU A 1
H34' H 0.7626 0.5629 0.9190 0.033 Uiso 0.34(2) 1 calc PR A 1
C35' C 0.608(3) 0.614(2) 0.922(2) 0.024(7) Uani 0.34(2) 1 d PU A 1
C36' C 0.532(4) 0.658(3) 0.885(3) 0.024(7) Uani 0.34(2) 1 d PU A 1
H36' H 0.4598 0.6728 0.9019 0.029 Uiso 0.34(2) 1 calc PR A 1

C37' C 0.561(6) 0.679(4) 0.822(4) 0.019(8) Uani 0.34(2) 1 d PU A 1
H37' H 0.5039 0.7069 0.7946 0.023 Uiso 0.34(2) 1 calc PR A 1
C38' C 0.572(3) 0.5903(12) 0.9882(14) 0.031(6) Uani 0.34(2) 1 d PU A 1
H38' H 0.4953 0.6064 0.9999 0.037 Uiso 0.34(2) 1 calc PR A 1
C39' C 0.632(2) 0.5494(10) 1.0333(11) 0.028(6) Uani 0.34(2) 1 d PU A 1
H39' H 0.7099 0.5333 1.0235 0.033 Uiso 0.34(2) 1 calc PR A 1
C40' C 0.588(3) 0.5274(15) 1.0979(15) 0.024(6) Uani 0.34(2) 1 d PU A 1
C41' C 0.475(3) 0.5466(16) 1.1137(18) 0.027(6) Uani 0.34(2) 1 d PU A 1
H41' H 0.4237 0.5752 1.0844 0.032 Uiso 0.34(2) 1 calc PR A 1
C42' C 0.436(8) 0.523(5) 1.174(4) 0.037(10) Uani 0.34(2) 1 d PU A 1
H42' H 0.3567 0.5363 1.1835 0.044 Uiso 0.34(2) 1 calc PR A 1
N7' N 0.504(11) 0.482(4) 1.220(5) 0.041(14) Uani 0.34(2) 1 d PU A 1
C43' C 0.615(4) 0.4660(19) 1.200(2) 0.041(9) Uani 0.34(2) 1 d PU A 1
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P1 P 0.37509(17) 0.82261(8) 0.92954(9) 0.0277(4) Uani 1 1 d D . .
F11 F 0.3798(4) 0.86930(19) 0.9945(2) 0.0378(10) Uani 1 1 d D . .
F12 F 0.2700(4) 0.8647(2) 0.8839(2) 0.0437(11) Uani 1 1 d D . .
F13 F 0.2650(4) 0.78394(19) 0.9569(2) 0.0400(10) Uani 1 1 d D . .
F14 F 0.3707(4) 0.7758(2) 0.8650(2) 0.0395(10) Uani 1 1 d D . .
F15 F 0.4780(4) 0.7803(2) 0.9756(2) 0.0419(10) Uani 1 1 d D . .
F16 F 0.4839(4) 0.8616(2) 0.9019(2) 0.0438(11) Uani 1 1 d D . .
P2 P 0.9773(5) 0.4520(3) 0.8325(3) 0.0302(14) Uani 0.684(18) 1 d PD B 1
F21 F 0.8678(11) 0.4414(5) 0.8771(5) 0.041(3) Uani 0.684(18) 1 d PD B 1
F22 F 0.9930(8) 0.5212(4) 0.8567(6) 0.058(3) Uani 0.684(18) 1 d PD B 1
F23 F 1.0789(11) 0.4319(6) 0.8967(7) 0.070(4) Uani 0.684(18) 1 d PD B 1
F24 F 1.0850(7) 0.4646(7) 0.7867(5) 0.066(4) Uani 0.684(18) 1 d PD B 1
F25 F 0.8731(8) 0.4769(8) 0.7685(4) 0.083(5) Uani 0.684(18) 1 d PD B 1
F26 F 0.9591(11) 0.3853(5) 0.8015(9) 0.113(7) Uani 0.684(18) 1 d PD B 1
P2' P 0.9772(16) 0.4338(8) 0.8464(8) 0.049(4) Uani 0.316(18) 1 d PD B 2
F21' F 0.852(3) 0.4464(16) 0.8818(16) 0.103(17) Uani 0.316(18) 1 d PD B 2
F22' F 0.981(3) 0.4957(11) 0.8291(17) 0.16(2) Uani 0.316(18) 1 d PD B 2
F23' F 1.064(2) 0.4454(13) 0.9208(10) 0.068(9) Uani 0.316(18) 1 d PD B 2
F24' F 1.1041(14) 0.4210(11) 0.8144(11) 0.061(7) Uani 0.316(18) 1 d PD B 2
F25' F 0.8926(16) 0.4295(11) 0.7710(8) 0.055(6) Uani 0.316(18) 1 d PD B 2
F26' F 0.9665(15) 0.3694(7) 0.8594(12) 0.059(6) Uani 0.316(18) 1 d PD B 2
C C 0.1449(18) 0.6304(8) 0.9636(8) 0.132(7) Uani 1 1 d . . .
H0A H 0.1379 0.5855 0.9577 0.198 Uiso 1 1 calc R . .
H0B H 0.0905 0.6508 0.9246 0.198 Uiso 1 1 calc R . .
H0C H 0.1184 0.6423 1.0078 0.198 Uiso 1 1 calc R . .
O O 0.2731(10) 0.6489(6) 0.9644(9) 0.140(5) Uani 1 1 d . . .
H0 H 0.2751 0.6861 0.9523 0.210 Uiso 1 1 calc R . .

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N2 0.022(3) 0.021(3) 0.019(2) -0.001(2) 0.003(2) 0.001(2)
N3 0.024(3) 0.025(3) 0.023(3) -0.003(2) 0.000(2) 0.003(2)
N4 0.022(3) 0.027(3) 0.023(3) -0.001(2) -0.001(2) 0.004(2)
N5 0.019(3) 0.027(3) 0.025(3) -0.006(2) -0.002(2) 0.003(2)
C1 0.026(4) 0.031(4) 0.030(3) -0.006(3) -0.002(3) -0.002(3)
C2 0.025(4) 0.035(4) 0.045(4) -0.006(3) -0.005(3) -0.003(3)
C3 0.022(3) 0.033(4) 0.052(4) -0.003(3) 0.000(3) 0.004(3)
C4 0.025(3) 0.026(3) 0.037(4) -0.003(3) 0.002(3) 0.001(3)
C5 0.023(3) 0.021(3) 0.026(3) 0.001(2) 0.002(2) -0.001(3)
C6 0.022(3) 0.022(3) 0.021(3) 0.000(2) 0.002(2) 0.000(2)
C7 0.021(3) 0.022(3) 0.026(3) -0.002(2) 0.004(2) 0.002(2)
C8 0.023(3) 0.022(3) 0.021(3) 0.001(2) 0.004(2) 0.000(2)
C9 0.021(3) 0.024(3) 0.021(3) 0.000(2) 0.001(2) 0.000(2)
C10 0.021(3) 0.022(3) 0.020(3) 0.001(2) 0.002(2) 0.001(2)
C11 0.025(3) 0.026(3) 0.022(3) -0.003(2) 0.001(2) 0.002(3)
C12 0.028(4) 0.030(4) 0.034(4) -0.008(3) -0.002(3) 0.003(3)
C13 0.025(4) 0.044(5) 0.049(4) -0.013(3) -0.006(3) 0.009(3)
C14 0.034(4) 0.037(4) 0.049(5) -0.014(3) -0.003(3) 0.013(3)
C15 0.032(4) 0.029(4) 0.033(4) -0.009(3) -0.001(3) 0.005(3)
C16 0.025(3) 0.023(3) 0.022(3) 0.000(2) 0.004(2) 0.000(3)
C17 0.023(3) 0.026(3) 0.024(3) -0.002(2) 0.002(3) 0.001(3)
C18 0.027(3) 0.024(3) 0.025(3) -0.002(2) 0.005(3) 0.003(3)
C19 0.032(4) 0.022(3) 0.029(3) -0.002(3) 0.004(3) -0.001(3)
C20 0.023(4) 0.028(4) 0.052(4) -0.008(3) 0.002(3) -0.004(3)
C21 0.024(3) 0.022(3) 0.047(4) -0.007(3) 0.004(3) 0.001(3)
C22 0.034(4) 0.028(4) 0.043(4) -0.010(3) 0.004(3) -0.002(3)
C23 0.035(4) 0.029(4) 0.025(3) -0.002(3) 0.003(3) 0.003(3)
C24 0.048(4) 0.030(4) 0.032(4) 0.005(3) 0.006(3) 0.004(3)
C25 0.051(5) 0.040(4) 0.027(4) 0.007(3) 0.006(3) 0.010(4)
C26 0.038(4) 0.037(4) 0.021(3) -0.007(3) -0.002(3) 0.011(3)
C27 0.022(3) 0.032(4) 0.022(3) -0.004(2) -0.003(2) 0.010(3)
C28 0.018(3) 0.033(4) 0.024(3) -0.006(3) -0.004(2) 0.006(3)
C29 0.028(4) 0.037(4) 0.026(3) -0.010(3) -0.002(3) 0.005(3)
C30 0.025(4) 0.035(4) 0.038(4) -0.016(3) -0.003(3) 0.002(3)
C31 0.024(4) 0.030(4) 0.044(4) -0.010(3) 0.001(3) -0.003(3)
C32 0.027(4) 0.030(4) 0.031(4) -0.005(3) 0.002(3) -0.001(3)
N6 0.027(14) 0.014(9) 0.019(7) -0.004(6) 0.002(8) 0.003(8)
C33 0.022(15) 0.027(9) 0.030(10) 0.003(6) -0.002(10) 0.007(10)
C34 0.022(11) 0.029(11) 0.028(8) -0.003(6) 0.005(9) 0.008(8)
C35 0.026(11) 0.017(8) 0.023(6) -0.003(5) -0.002(7) 0.005(7)
C36 0.028(10) 0.017(7) 0.025(6) 0.002(4) 0.003(7) 0.008(7)
C37 0.030(13) 0.017(8) 0.030(8) 0.000(6) 0.005(7) 0.009(7)
C38 0.036(8) 0.025(6) 0.026(6) -0.001(4) 0.001(5) 0.000(5)
C39 0.039(7) 0.030(6) 0.026(6) 0.000(4) 0.003(5) 0.000(5)
```

C40 0.023(9) 0.030(7) 0.027(6) -0.005(5) 0.002(5) -0.005(6)
C41 0.024(7) 0.047(10) 0.025(7) 0.002(7) 0.000(5) -0.003(7)
C42 0.031(7) 0.048(11) 0.028(7) 0.000(7) -0.001(5) 0.002(9)
N7 0.029(9) 0.030(12) 0.024(7) 0.005(8) 0.004(6) -0.004(9)
C43 0.035(12) 0.026(9) 0.021(9) 0.000(7) 0.004(7) -0.001(8)
C44 0.030(9) 0.029(7) 0.026(6) 0.001(5) 0.004(7) 0.002(7)
N6' 0.023(19) 0.02(3) 0.031(19) -0.007(16) 0.005(13) -0.001(16)
C33' 0.013(19) 0.033(19) 0.038(15) -0.023(13) 0.003(12) 0.004(13)
C34' 0.018(18) 0.024(14) 0.039(14) -0.003(11) 0.001(12) 0.015(13)
C35' 0.015(17) 0.026(16) 0.027(11) 0.002(9) -0.004(12) 0.014(13)
C36' 0.015(19) 0.024(16) 0.031(13) 0.008(10) 0.000(14) 0.011(13)
C37' 0.01(2) 0.023(19) 0.021(15) -0.001(10) -0.003(15) 0.004(14)
C38' 0.035(15) 0.029(12) 0.027(12) -0.001(9) 0.002(11) 0.001(11)
C39' 0.030(12) 0.028(12) 0.025(10) 0.000(8) 0.002(8) -0.003(9)
C40' 0.019(14) 0.029(14) 0.022(11) -0.004(9) -0.003(10) 0.005(10)
C41' 0.010(16) 0.033(14) 0.033(13) -0.004(11) -0.009(11) 0.000(13)
C42' 0.03(2) 0.035(19) 0.04(3) -0.016(17) 0.009(14) -0.009(15)
N7' 0.034(18) 0.05(4) 0.036(16) -0.004(18) -0.002(12) 0.00(2)
C43' 0.039(14) 0.05(2) 0.033(13) 0.006(15) 0.003(10) 0.000(17)
C44' 0.032(12) 0.031(14) 0.026(11) 0.007(10) -0.008(9) 0.003(10)
P1 0.0258(9) 0.0286(9) 0.0282(9) -0.0073(7) 0.0025(7) -0.0007(7)
F11 0.043(2) 0.034(2) 0.038(2) -0.0126(18) 0.0077(19) -0.0017(19)
F12 0.040(3) 0.039(2) 0.048(3) 0.0017(19) -0.007(2) 0.005(2)
F13 0.038(2) 0.035(2) 0.049(3) -0.0061(19) 0.014(2) -0.0070(19)
F14 0.037(2) 0.045(2) 0.036(2) -0.0190(19) 0.0038(18) -0.004(2)
F15 0.038(2) 0.041(2) 0.043(2) -0.0080(19) -0.0058(19) 0.010(2)
F16 0.040(3) 0.051(3) 0.042(2) -0.009(2) 0.010(2) -0.014(2)
P2 0.0236(18) 0.033(3) 0.035(3) -0.002(2) 0.0093(16) -0.0018(19)
F21 0.028(5) 0.054(6) 0.043(6) 0.006(4) 0.014(4) -0.002(4)
F22 0.051(5) 0.024(4) 0.099(7) 0.011(4) 0.011(5) -0.008(4)
F23 0.029(5) 0.084(8) 0.092(9) 0.059(7) -0.006(6) 0.001(5)
F24 0.036(4) 0.115(10) 0.052(5) -0.012(5) 0.017(4) -0.014(5)
F25 0.042(5) 0.160(14) 0.044(5) 0.022(6) -0.004(4) -0.002(7)
F26 0.090(8) 0.080(9) 0.189(18) -0.091(11) 0.085(11) -0.037(7)
P2' 0.048(6) 0.051(9) 0.048(7) 0.011(5) 0.011(5) -0.011(6)
F21' 0.049(19) 0.16(4) 0.10(3) 0.02(2) 0.027(18) 0.03(2)
F22' 0.15(3) 0.07(3) 0.21(4) 0.01(3) -0.08(3) -0.04(2)
F23' 0.047(15) 0.102(19) 0.050(13) -0.023(12) -0.006(10) -0.013(12)
F24' 0.047(10) 0.076(16) 0.064(13) -0.028(11) 0.019(9) -0.007(10)
F25' 0.053(11) 0.064(14) 0.044(9) -0.001(9) -0.006(8) 0.006(10)
F26' 0.028(9) 0.057(11) 0.087(15) -0.001(10) -0.007(9) -0.011(7)
C 0.167(17) 0.138(14) 0.089(10) 0.028(10) 0.015(11) -0.072(13)
O 0.097(8) 0.115(9) 0.216(14) 0.036(9) 0.051(8) 0.014(7)

_geom_special_details
;
All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles

 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
;

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Ru N6 2.10(4) . ?
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N2 C6 1.351(8) . ?
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C7 H7 0.9500 . ?
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P2 F26 1.562(10) . ?
P2 F24 1.593(9) . ?
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P2' F23' 1.60(2) . ?

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H22A C22 H22C 109.5 . . ?
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F21' P2' F22' 89.8(15) . . ?
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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C2"' C 0.5144(3) 0.6081(4) 0.12240(5) 0.0292(7) Uani 1 1 d . . .
H2"' H 0.4361 0.5752 0.1199 0.035 Uiso 1 1 calc R . .
C3"' C 0.5693(3) 0.6985(4) 0.10536(5) 0.0318(8) Uani 1 1 d . . .
H3"' H 0.5277 0.7262 0.0913 0.038 Uiso 1 1 calc R . .
C4"' C 0.6850(3) 0.7500(4) 0.10839(5) 0.0285(7) Uani 1 1 d . . .
C5"' C 0.7446(3) 0.7094(4) 0.12897(5) 0.0316(8) Uani 1 1 d . . .
H5"' H 0.8228 0.7431 0.1314 0.038 Uiso 1 1 calc R . .
C6"' C 0.6895(3) 0.6191(4) 0.14597(5) 0.0289(7) Uani 1 1 d . . .
H6"' H 0.7310 0.5923 0.1601 0.035 Uiso 1 1 calc R . .
C7 C 0.7458(3) 0.8439(4) 0.08946(6) 0.0378(8) Uani 1 1 d . . .
H7A H 0.6950 0.8543 0.0752 0.045 Uiso 1 1 calc R . .
H7B H 0.7635 0.9478 0.0956 0.045 Uiso 1 1 calc R . .
```

C01 C 0.9134(3) 0.8409(4) 0.06485(5) 0.0359(8) Uani 1 1 d . . .
H01A H 0.9382 0.9448 0.0699 0.043 Uiso 1 1 calc R . .
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C02 C 1.0214(3) 0.7378(4) 0.05998(5) 0.0298(7) Uani 1 1 d . . .
C03 C 0.8933(3) 0.4204(5) 0.03105(6) 0.0517(10) Uani 1 1 d . . .
H03 H 0.8373 0.3768 0.0205 0.062 Uiso 1 1 calc R . .
C04 C 0.9888(3) 0.3375(5) 0.04033(6) 0.0454(9) Uani 1 1 d . . .
H04 H 1.0080 0.2327 0.0378 0.054 Uiso 1 1 calc R . .
C05 C 1.0474(3) 0.4434(4) 0.05386(6) 0.0364(8) Uani 1 1 d . . .
H05 H 1.1186 0.4267 0.0622 0.044 Uiso 1 1 calc R . .
C03' C 1.1338(3) 0.9388(5) 0.01154(6) 0.0492(10) Uani 1 1 d . . .
H03' H 1.1300 1.0142 -0.0007 0.059 Uiso 1 1 calc R . .
C04' C 1.2248(3) 0.8356(5) 0.01366(6) 0.0454(10) Uani 1 1 d . . .
H04' H 1.2922 0.8277 0.0038 0.054 Uiso 1 1 calc R . .
C05' C 1.1972(3) 0.7474(5) 0.03295(6) 0.0472(10) Uani 1 1 d . . .
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C03" C 1.2161(3) 0.7966(4) 0.10731(6) 0.0396(9) Uani 1 1 d . . .
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C05" C 1.0676(3) 0.6375(4) 0.10095(5) 0.0377(9) Uani 1 1 d . . .
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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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N01' N02' 1.354(4) . ?
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C6"' C1"' C2"' C3"' 0.7(5) . . . . ?
C4' C1"' C2"' C3"' 178.3(3) . . . . ?
C1"' C2"' C3"' C4"' -0.2(5) . . . . ?
C2"' C3"' C4"' C5"' -0.2(5) . . . . ?
C2"' C3"' C4"' C7 178.0(3) . . . . ?
C3"' C4"' C5"' C6"' 0.2(5) . . . . ?
C7 C4"' C5"' C6"' -178.0(3) . . . . ?
C4"' C5"' C6"' C1"' 0.3(5) . . . . ?
C2"' C1"' C6"' C5"' -0.8(5) . . . . ?
C4' C1"' C6"' C5"' -178.3(3) . . . . ?
C01 O C7 C4"' 177.4(3) . . . . ?
C5"' C4"' C7 O 53.9(4) . . . . ?
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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C5" C 0.6170(6) -0.2186(2) 1.0549(3) 0.0697(15) Uani 1 1 d . . .
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C6" C 0.6306(6) -0.1858(2) 1.1213(3) 0.0642(14) Uani 1 1 d . . .
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C5"' C -0.0287(6) 0.1488(2) 0.9141(3) 0.0702(15) Uani 1 1 d . . .
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C4"' C -0.0760(5) 0.1203(2) 0.8458(3) 0.0492(12) Uani 1 1 d . . .
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C2"' C 0.0708(5) 0.0333(2) 0.8955(3) 0.0484(12) Uani 1 1 d . . .
H2"' H 0.1036 -0.0061 0.8880 0.058 Uiso 1 1 calc R . .
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H7B H -0.1807 0.1953 0.7931 0.075 Uiso 1 1 calc R . .
C01 C 0.0249(5) 0.16921(19) 0.7022(2) 0.0449(11) Uani 1 1 d . . .
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C4"' 0.052(3) 0.057(4) 0.040(3) 0.014(3) 0.010(2) 0.015(2)
C3"' 0.059(3) 0.065(4) 0.038(3) 0.007(3) 0.003(2) 0.001(3)
C2"' 0.060(3) 0.044(3) 0.041(3) 0.008(2) 0.004(2) 0.005(2)
C7 0.056(3) 0.088(4) 0.045(3) 0.018(3) 0.012(2) 0.017(3)
C01 0.049(3) 0.046(3) 0.039(3) 0.001(2) 0.003(2) 0.000(2)
C02 0.045(3) 0.048(3) 0.035(3) 0.007(2) 0.000(2) 0.000(2)
C03 0.097(4) 0.046(4) 0.060(4) 0.004(3) 0.005(3) 0.009(3)
C04 0.097(4) 0.042(4) 0.086(4) -0.006(3) -0.002(3) -0.014(3)
C05 0.061(3) 0.071(4) 0.073(4) -0.010(3) -0.014(3) -0.002(3)
C03' 0.106(5) 0.075(4) 0.036(3) 0.009(3) -0.009(3) 0.000(3)
C04' 0.069(4) 0.064(4) 0.062(4) 0.024(3) -0.011(3) 0.003(3)
C05' 0.053(3) 0.050(3) 0.056(3) 0.013(3) 0.000(2) 0.000(2)
C03" 0.059(4) 0.112(6) 0.089(5) 0.053(4) -0.020(3) -0.035(4)
C04" 0.056(4) 0.124(6) 0.117(6) 0.047(5) 0.023(4) -0.005(4)
C05" 0.059(4) 0.104(5) 0.098(5) 0.006(4) 0.027(3) -0.003(4)
B 0.090(6) 0.082(7) 0.058(6) 0.009(5) -0.015(5) 0.016(5)
F1A 0.133(11) 0.086(8) 0.201(16) -0.015(9) 0.075(9) 0.046(7)
F2A 0.239(15) 0.220(17) 0.080(7) 0.057(7) 0.092(9) 0.102(12)
F3A 0.135(10) 0.257(15) 0.053(8) -0.004(7) -0.026(5) -0.014(7)
F4A 0.168(12) 0.153(17) 0.219(19) 0.075(13) -0.074(10) -0.051(10)
F1B 0.63(3) 0.169(13) 0.042(7) -0.007(7) -0.046(11) -0.003(16)
F2B 0.150(10) 0.075(7) 0.100(7) 0.031(5) -0.030(6) 0.029(6)
F3B 0.337(19) 0.037(8) 0.092(9) -0.042(6) -0.067(10) 0.058(9)
F4B 0.117(9) 0.226(16) 0.45(3) -0.120(19) -0.069(15) 0.069(9)

_geom_special_details

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;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Ag N1" 2.372(4) . ?
Ag N1 2.586(4) . ?
N1 C2 1.302(6) . ?
N1 C6 1.340(7) . ?
N1' C2' 1.342(5) . ?
N1' C6' 1.350(5) . ?
N1" C6" 1.342(5) . ?
N1" C2" 1.348(5) . ?
N01 C05 1.343(5) . ?
N01 N02 1.349(4) . ?
N01 C02 1.478(5) . ?
N02 C03 1.339(6) . ?
N02 Ag 2.236(4) 3_657 ?
N01' C05' 1.340(5) . ?
N01' N02' 1.361(5) . ?
N01' C02 1.456(5) . ?
N02' C03' 1.331(6) . ?
N01" C05" 1.342(6) . ?
N01" N02" 1.354(5) . ?
N01" C02 1.446(5) . ?
N02" C03" 1.340(7) . ?
O C01 1.411(4) . ?
O C7 1.438(5) . ?
C2 C3 1.361(7) . ?
C2 C2' 1.497(6) . ?
C3 C4 1.376(8) . ?
C3 H3 0.9300 . ?
C4 C5 1.338(8) . ?
C4 H4 0.9300 . ?
C5 C6 1.336(8) . ?
C5 H5 0.9300 . ?
C6 H6 0.9300 . ?
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C2' C3' 1.378(5) . ?
C3' C4' 1.391(5) . ?
C3' H3' 0.9300 . ?
C4' C5' 1.390(6) . ?
C4' C1''' 1.493(5) . ?
C5' C6' 1.389(5) . ?
C5' H5' 0.9300 . ?
C6' C2" 1.497(6) . ?
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C3" C4" 1.377(6) . ?
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C4" C5" 1.362(6) . ?
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C5" H5" 0.9300 . ?
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C7 H7A 0.9700 . ?
C7 H7B 0.9700 . ?
C01 C02 1.529(5) . ?
C01 H01A 0.9700 . ?
C01 H01B 0.9700 . ?
C03 C04 1.373(6) . ?
C03 H03 0.9300 . ?
C04 C05 1.365(6) . ?
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C05 H05 0.9300 . ?
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C04' H04' 0.9300 . ?
C05' H05' 0.9300 . ?
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C04" H04" 0.9300 . ?
C05" H05" 0.9300 . ?
B F4A 1.315(2) . ?
B F4B 1.315(2) . ?

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B F1B 1.316(2) . ?
B F3A 1.316(2) . ?
B F1A 1.316(2) . ?
B F2B 1.316(2) . ?
B F3B 1.316(2) . ?
B F2A 1.316(2) . ?

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N02 Ag N1 89.31(14) 3_657 . ?
N1' Ag N1 66.16(14) . . ?
N1" Ag N1 133.25(14) . . ?
C2 N1 C6 117.2(5) . . ?
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C2" N1" Ag 116.1(3) . . ?
C05 N01 N02 111.7(4) . . ?
C05 N01 C02 127.5(4) . . ?
N02 N01 C02 119.6(3) . . ?
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N01 N02 Ag 133.3(3) . 3_657 ?
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C05' N01' C02 129.8(4) . . ?
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C03" N02" N01" 103.2(5) . . ?
C01 O C7 112.5(3) . . ?
N1 C2 C3 121.5(5) . . ?
N1 C2 C2' 117.3(5) . . ?
C3 C2 C2' 121.1(5) . . ?
C2 C3 C4 119.5(6) . . ?
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C4 C3 H3 120.3 . . ?
C5 C4 C3 119.3(7) . . ?
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C3 C4 H4 120.3 . . ?
C4 C5 C6 117.3(6) . . ?
C4 C5 H5 121.3 . . ?
C6 C5 H5 121.3 . . ?
C5 C6 N1 124.9(6) . . ?
C5 C6 H6 117.5 . . ?
N1 C6 H6 117.5 . . ?
N1' C2' C3' 122.0(4) . . ?
N1' C2' C2 116.2(4) . . ?
C3' C2' C2 121.8(4) . . ?
C2' C3' C4' 120.9(4) . . ?
C2' C3' H3' 119.5 . . ?
C4' C3' H3' 119.5 . . ?
C5' C4' C3' 116.6(4) . . ?
C5' C4' C1''' 121.2(4) . . ?
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N1' C6' C5' 122.1(4) . . ?
N1' C6' C2" 116.0(4) . . ?
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C2" C3" C4" 120.3(4) . . ?
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C4" C3" H3" 119.9 . . ?
C5" C4" C3" 119.1(5) . . ?
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C3" C4" H4" 120.4 . . ?
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C6" C5" H5" 121.0 . . ?
C4" C5" H5" 121.0 . . ?
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C5''' C4''' C7 121.0(5) . . ?
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O C7 C4''' 113.0(4) . . ?
O C7 H7A 109.0 . . ?
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O C7 H7B 109.0 . . ?
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H7A C7 H7B 107.8 . . ?
O C01 C02 108.5(3) . . ?
O C01 H01A 110.0 . . ?
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O C01 H01B 110.0 . . ?
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N01'' C02 N01 107.4(3) . . ?
N01' C02 N01 107.4(3) . . ?
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N01' C02 C01 111.7(3) . . ?
N01 C02 C01 109.4(3) . . ?
N02 C03 C04 111.7(5) . . ?
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C05 C04 C03 105.2(5) . . ?
C05 C04 H04 127.4 . . ?
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N01 C05 C04 107.1(5) . . ?
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C04 C05 H05 126.4 . . ?
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C05' C04' C03' 105.3(4) . . ?
C05' C04' H04' 127.3 . . ?
C03' C04' H04' 127.3 . . ?
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C04' C05' H05' 126.7 . . ?
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N02'' C03'' H03'' 123.9 . . ?
C04'' C03'' H03'' 123.9 . . ?

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C05" C04" H04" 127.0 . . ?
C03" C04" H04" 127.0 . . ?
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N01" C05" H05" 126.5 . . ?
C04" C05" H05" 126.5 . . ?
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F4A B F1B 92.9(8) . . ?
F4B B F1B 109.49(10) . . ?
F4A B F3A 109.50(10) . . ?
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F1B B F3A 37.2(7) . . ?
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F1B B F1A 85.3(8) . . ?
F3A B F1A 109.47(10) . . ?
F4A B F2B 102.9(10) . . ?
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N1" Ag N1 C6 159.8(5) . . . . ?
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N1 Ag N1" C2" 34.4(4) . . . . ?
C05 N01 N02 C03 1.4(5) . . . . ?
C02 N01 N02 C03 169.5(4) . . . . ?
C05 N01 N02 Ag -179.1(3) . . . 3_657 ?
C02 N01 N02 Ag -11.0(5) . . . 3_657 ?
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Ag N1 C2 C3 -166.8(5) . . . . ?
C6 N1 C2 C2' -179.7(5) . . . . ?
Ag N1 C2 C2' 15.2(6) . . . . ?
N1 C2 C3 C4 1.0(11) . . . . ?
C2' C2 C3 C4 178.9(7) . . . . ?
C2 C3 C4 C5 2.4(13) . . . . ?
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C4 C5 C6 N1 4.3(12) . . . . ?
C2 N1 C6 C5 -1.0(11) . . . . ?
Ag N1 C6 C5 162.2(6) . . . . ?
C6' N1' C2' C3' -2.7(6) . . . . ?
Ag N1' C2' C3' 172.1(3) . . . . ?
C6' N1' C2' C2 176.7(4) . . . . ?
Ag N1' C2' C2 -8.5(5) . . . . ?
N1 C2 C2' N1' -5.9(6) . . . . ?
C3 C2 C2' N1' 176.1(5) . . . . ?
N1 C2 C2' C3' 173.5(5) . . . . ?
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N1' C2' C3' C4' 0.2(7) . . . . ?
C2 C2' C3' C4' -179.1(4) . . . . ?
C2' C3' C4' C5' 1.3(6) . . . . ?
C2' C3' C4' C1"' -178.7(4) . . . . ?
C3' C4' C5' C6' -0.4(6) . . . . ?
C1"' C4' C5' C6' 179.7(4) . . . . ?
C2' N1' C6' C5' 3.7(6) . . . . ?

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Ag N1' C6' C5' -171.4(3) . . . . ?
C2' N1' C6' C2" -176.1(4) . . . . ?
Ag N1' C6' C2" 8.9(4) . . . . ?
C4' C5' C6' N1' -2.1(6) . . . . ?
C4' C5' C6' C2" 177.6(4) . . . . ?
C6" N1" C2" C3" -0.3(6) . . . . ?
Ag N1" C2" C3" 167.8(3) . . . . ?
C6" N1" C2" C6' 179.4(4) . . . . ?
Ag N1" C2" C6' -12.6(5) . . . . ?
N1' C6' C2" N1" 2.7(5) . . . . ?
C5' C6' C2" N1" -177.0(4) . . . . ?
N1' C6' C2" C3" -177.6(4) . . . . ?
C5' C6' C2" C3" 2.6(6) . . . . ?
N1" C2" C3" C4" -1.1(7) . . . . ?
C6' C2" C3" C4" 179.3(4) . . . . ?
C2" C3" C4" C5" 1.4(8) . . . . ?
C3" C4" C5" C6" -0.4(8) . . . . ?
C2" N1" C6" C5" 1.4(7) . . . . ?
Ag N1" C6" C5" -165.5(4) . . . . ?
C4" C5" C6" N1" -1.0(8) . . . . ?
C5' C4' C1"' C6"' -170.7(4) . . . . ?
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C5' C4' C1"' C2"' 8.7(6) . . . . ?
C3' C4' C1"' C2"' -171.2(4) . . . . ?
C2"' C1"' C6"' C5"' -1.4(7) . . . . ?
C4' C1"' C6"' C5"' 178.1(4) . . . . ?
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C6"' C5"' C4"' C3"' 0.4(7) . . . . ?
C6"' C5"' C4"' C7 -179.3(4) . . . . ?
C5"' C4"' C3"' C2"' -1.2(6) . . . . ?
C7 C4"' C3"' C2"' 178.4(4) . . . . ?
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C4' C1"' C2"' C3"' -178.9(4) . . . . ?
C01 O C7 C4"' -63.4(5) . . . . ?
C5"' C4"' C7 O 134.4(4) . . . . ?
C3"' C4"' C7 O -45.2(6) . . . . ?
C7 O C01 C02 164.5(3) . . . . ?
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N02" N01" C02 N01 -139.7(4) . . . . ?
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N02" N01" C02 C01 -20.5(5) . . . . ?
C05' N01' C02 N01" -133.1(5) . . . . ?
N02' N01' C02 N01" 54.4(5) . . . . ?
C05' N01' C02 N01 110.1(5) . . . . ?
N02' N01' C02 N01 -62.4(5) . . . . ?
C05' N01' C02 C01 -9.9(6) . . . . ?
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N02' N01' C02 C01 177.6(4) . . . . ?
C05 N01 C02 N01" -144.3(4) . . . . ?
N02 N01 C02 N01" 49.7(5) . . . . ?
C05 N01 C02 N01' -25.4(6) . . . . ?
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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 on F^2^ are statistically about twice as large as those based on F, and R-
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O43 O 0.4695(2) 0.54715(18) 1.21298(11) 0.0502(5) Uani 1 1 d . . .
N5 N 0.8926(2) 0.83848(15) 1.07363(11) 0.0291(4) Uani 1 1 d . . .
O51 O 0.85186(18) 0.76544(14) 1.12644(9) 0.0337(4) Uani 1 1 d . . .
O53 O 1.01959(18) 0.87568(14) 1.07026(11) 0.0390(4) Uani 1 1 d . . .
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C05 C 0.1605(4) 0.7865(3) 0.31140(16) 0.0523(7) Uani 1 1 d . . .
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N02 N -0.0734(3) 0.7863(2) 0.38564(13) 0.0487(6) Uani 1 1 d . . .
N01' N 0.2885(3) 0.8012(2) 0.44769(14) 0.0489(6) Uani 1 1 d . . .
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C2' C 0.4303(2) 0.82983(17) 1.03384(12) 0.0246(4) Uani 1 1 d . . .
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C4' C 0.3895(2) 0.78336(18) 0.90581(12) 0.0254(4) Uani 1 1 d . . .
C5' C 0.4962(2) 0.68540(18) 0.90818(12) 0.0255(4) Uani 1 1 d . . .
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C2" C 0.6762(2) 0.56396(17) 0.98677(12) 0.0249(4) Uani 1 1 d . . .
C3" C 0.7306(2) 0.47735(19) 0.93310(13) 0.0289(5) Uani 1 1 d . . .
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H7B H 0.1120 0.9546 0.6142 0.071 Uiso 1 1 calc R . .
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C01 C 0.0274(3) 0.8094(3) 0.52611(16) 0.0507(7) Uani 1 1 d . . .
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H8B H 1.1016 0.4956 0.7046 0.121 Uiso 0.50 1 calc PR A 1
H8C H 0.9375 0.4676 0.6939 0.121 Uiso 0.50 1 calc PR A 1
C9 C 0.928(2) 0.5982(16) 0.7674(10) 0.074(5) Uani 0.50 1 d PD A 1
N6 N 0.8768(14) 0.6731(7) 0.8012(8) 0.124(5) Uani 0.50 1 d PD A 1
C8' C 0.9979(5) 0.4882(4) 0.7309(2) 0.0809(11) Uani 0.50 1 d P B 2
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H8'C H 0.9367 0.4255 0.7395 0.121 Uiso 0.50 1 calc PR B 2
C9' C 0.8952(18) 0.5967(14) 0.7422(9) 0.063(4) Uani 0.50 1 d PD B 2
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C4" 0.0271(11) 0.0251(11) 0.0379(13) -0.0034(9) -0.0021(9) 0.0027(9)
C5" 0.0249(11) 0.0270(11) 0.0418(13) 0.0039(9) -0.0053(9) 0.0023(9)
C6" 0.0270(11) 0.0326(12) 0.0334(12) 0.0043(9) -0.0100(9) 0.0000(9)
C2 0.0239(10) 0.0259(11) 0.0262(11) -0.0015(8) -0.0029(8) -0.0037(8)
C3 0.0333(12) 0.0281(11) 0.0312(12) 0.0000(9) -0.0044(9) -0.0005(9)
C4 0.0429(13) 0.0262(11) 0.0370(13) -0.0049(10) -0.0009(10) -0.0003(10)
```

C5 0.0448(14) 0.0381(13) 0.0314(12) -0.0104(10) -0.0051(10) -0.0057(11)
C6 0.0378(13) 0.0392(13) 0.0296(12) -0.0055(10) -0.0099(10) -0.0027(10)
C1''' 0.0282(11) 0.0258(11) 0.0285(11) 0.0009(9) -0.0069(9) -0.0017(8)
C2''' 0.0369(13) 0.0397(13) 0.0324(12) -0.0037(10) -0.0099(10) 0.0098(10)
C3''' 0.0499(15) 0.0492(15) 0.0296(13) -0.0084(11) -0.0120(11) 0.0108(12)
C4''' 0.0464(14) 0.0423(14) 0.0362(13) -0.0008(11) -0.0183(11) 0.0064(11)
C5''' 0.0375(13) 0.0374(13) 0.0414(14) -0.0016(11) -0.0140(11) 0.0098(10)
C6''' 0.0318(12) 0.0314(12) 0.0319(12) -0.0027(9) -0.0083(9) 0.0034(9)
C7 0.070(2) 0.0621(19) 0.0457(16) -0.0119(14) -0.0314(15) 0.0230(16)
O 0.0951(17) 0.0657(14) 0.0440(12) -0.0059(10) -0.0396(12) 0.0184(12)
C01 0.0545(17) 0.0614(18) 0.0376(14) 0.0030(13) -0.0189(12) 0.0034(14)
C02 0.0435(14) 0.0496(15) 0.0381(14) 0.0049(11) -0.0178(11) -0.0073(12)
C8 0.097(3) 0.079(3) 0.064(2) -0.0188(19) -0.018(2) 0.012(2)
C9 0.060(7) 0.056(10) 0.098(13) 0.027(9) -0.003(7) 0.005(6)
N6 0.098(7) 0.040(4) 0.204(13) 0.015(6) 0.045(8) 0.003(5)
C8' 0.097(3) 0.079(3) 0.064(2) -0.0188(19) -0.018(2) 0.012(2)
C9' 0.064(9) 0.048(8) 0.072(8) -0.022(6) 0.009(5) -0.014(7)
N6' 0.074(5) 0.064(5) 0.145(9) -0.043(5) 0.006(5) 0.005(4)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Cu O41 1.9849(16) . ?
Cu N1 2.0249(19) . ?
Cu N1" 2.0407(18) . ?
Cu O51 2.1990(16) . ?
N1' C6' 1.341(3) . ?
N1' C2' 1.347(3) . ?
N1" C6" 1.347(3) . ?
N1" C2" 1.352(3) . ?
N1 C6 1.346(3) . ?
N1 C2 1.353(3) . ?
N4 O42 1.220(3) . ?
N4 O43 1.239(3) . ?
N4 O41 1.298(3) . ?

N5 O52 1.231(3) . ?
N5 O53 1.243(2) . ?
N5 O51 1.275(2) . ?
N01 N02 1.337(3) . ?
N01 C05 1.356(4) . ?
N01 C02 1.458(3) . ?
C05 C04 1.358(4) . ?
C05 H05 0.9300 . ?
C04 C03 1.381(5) . ?
C04 H04 0.9300 . ?
C03 N02 1.325(4) . ?
C03 H03 0.9300 . ?
N01' C05' 1.346(4) . ?
N01' N02' 1.366(4) . ?
N01' C02 1.462(3) . ?
C05' C04' 1.359(5) . ?
C05' H05' 0.9300 . ?
C04' C03' 1.389(6) . ?
C04' H04' 0.9300 . ?
C03' N02' 1.334(4) . ?
C03' H03' 0.9300 . ?
N01" C05" 1.346(4) . ?
N01" N02" 1.363(4) . ?
N01" C02 1.453(4) . ?
C05" C04" 1.361(5) . ?
C05" H05" 0.9300 . ?
C04" C03" 1.375(5) . ?
C04" H04" 0.9300 . ?
C03" N02" 1.330(4) . ?
C03" H03" 0.9300 . ?
C2' C3' 1.379(3) . ?
C2' C2 1.486(3) . ?
C3' C4' 1.407(3) . ?
C3' H3' 0.9300 . ?
C4' C5' 1.407(3) . ?
C4' C1'" 1.483(3) . ?
C5' C6' 1.377(3) . ?
C5' H5' 0.9300 . ?
C6' C2" 1.491(3) . ?
C2" C3" 1.388(3) . ?
C3" C4" 1.389(3) . ?
C3" H3" 0.9300 . ?
C4" C5" 1.384(3) . ?
C4" H4" 0.9300 . ?
C5" C6" 1.379(3) . ?
C5" H5" 0.9300 . ?
C6" H6" 0.9300 . ?
C2 C3 1.386(3) . ?
C3 C4 1.390(3) . ?

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C3 H3 0.9300 . ?
C4 C5 1.382(4) . ?
C4 H4 0.9300 . ?
C5 C6 1.381(3) . ?
C5 H5 0.9300 . ?
C6 H6 0.9300 . ?
C1''' C2''' 1.402(3) . ?
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C5''' H5''' 0.9300 . ?
C6''' H6''' 0.9300 . ?
C7 O 1.371(4) . ?
C7 H7A 0.9700 . ?
C7 H7B 0.9700 . ?
O C01 1.410(3) . ?
C01 C02 1.529(4) . ?
C01 H01A 0.9700 . ?
C01 H01B 0.9700 . ?
C8 C9 1.48(2) . ?
C8 H8A 0.9600 . ?
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C9' N6' 1.106(18) . ?

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N1' Cu N1 80.07(7) . . ?
O41 Cu N1 99.52(7) . . ?
N1' Cu N1" 79.60(7) . . ?
O41 Cu N1" 99.10(7) . . ?
N1 Cu N1" 159.64(7) . . ?
N1' Cu O51 118.75(7) . . ?
O41 Cu O51 81.94(6) . . ?
N1 Cu O51 95.48(7) . . ?
N1" Cu O51 95.22(7) . . ?
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C6' N1' C2' 120.91(18) . . ?
C6' N1' Cu 119.99(14) . . ?
C2' N1' Cu 119.10(14) . . ?
C6" N1" C2" 118.79(19) . . ?
C6" N1" Cu 126.87(15) . . ?
C2" N1" Cu 114.32(13) . . ?
C6 N1 C2 118.68(19) . . ?
C6 N1 Cu 126.60(16) . . ?
C2 N1 Cu 114.60(14) . . ?
O42 N4 O43 123.7(2) . . ?
O42 N4 O41 118.3(2) . . ?
O43 N4 O41 117.98(19) . . ?
N4 O41 Cu 111.06(13) . . ?
O52 N5 O53 121.99(19) . . ?
O52 N5 O51 119.08(19) . . ?
O53 N5 O51 118.93(18) . . ?
N5 O51 Cu 115.78(13) . . ?
N02 N01 C05 111.9(2) . . ?
N02 N01 C02 120.3(2) . . ?
C05 N01 C02 127.5(2) . . ?
N01 C05 C04 106.9(3) . . ?
N01 C05 H05 126.6 . . ?
C04 C05 H05 126.6 . . ?
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C05 C04 H04 127.7 . . ?
C03 C04 H04 127.7 . . ?
N02 C03 C04 112.4(3) . . ?
N02 C03 H03 123.8 . . ?
C04 C03 H03 123.8 . . ?
C03 N02 N01 104.2(2) . . ?
C05' N01' N02' 112.9(3) . . ?
C05' N01' C02 129.7(3) . . ?
N02' N01' C02 117.3(2) . . ?
N01' C05' C04' 106.6(3) . . ?
N01' C05' H05' 126.7 . . ?
C04' C05' H05' 126.7 . . ?
C05' C04' C03' 105.2(3) . . ?
C05' C04' H04' 127.4 . . ?
C03' C04' H04' 127.4 . . ?
N02' C03' C04' 112.7(3) . . ?
N02' C03' H03' 123.6 . . ?
C04' C03' H03' 123.6 . . ?
C03' N02' N01' 102.6(3) . . ?
C05" N01" N02" 111.3(3) . . ?
C05" N01" C02 129.4(3) . . ?
N02" N01" C02 118.7(2) . . ?
N01" C05" C04" 107.3(3) . . ?
N01" C05" H05" 126.4 . . ?
C04" C05" H05" 126.4 . . ?

C05" C04" C03" 105.2(3) . . ?
C05" C04" H04" 127.4 . . ?
C03" C04" H04" 127.4 . . ?
N02" C03" C04" 112.3(3) . . ?
N02" C03" H03" 123.9 . . ?
C04" C03" H03" 123.9 . . ?
C03" N02" N01" 103.9(3) . . ?
N1' C2' C3' 120.88(19) . . ?
N1' C2' C2 112.23(18) . . ?
C3' C2' C2 126.88(19) . . ?
C2' C3' C4' 119.57(19) . . ?
C2' C3' H3' 120.2 . . ?
C4' C3' H3' 120.2 . . ?
C3' C4' C5' 117.96(19) . . ?
C3' C4' C1'" 120.45(19) . . ?
C5' C4' C1'" 121.59(19) . . ?
C6' C5' C4' 119.42(19) . . ?
C6' C5' H5' 120.3 . . ?
C4' C5' H5' 120.3 . . ?
N1' C6' C5' 121.26(18) . . ?
N1' C6' C2" 111.84(18) . . ?
C5' C6' C2" 126.90(19) . . ?
N1" C2" C3" 121.83(19) . . ?
N1" C2" C6' 114.19(18) . . ?
C3" C2" C6' 123.97(19) . . ?
C2" C3" C4" 118.7(2) . . ?
C2" C3" H3" 120.6 . . ?
C4" C3" H3" 120.6 . . ?
C5" C4" C3" 119.4(2) . . ?
C5" C4" H4" 120.3 . . ?
C3" C4" H4" 120.3 . . ?
C6" C5" C4" 118.9(2) . . ?
C6" C5" H5" 120.5 . . ?
C4" C5" H5" 120.5 . . ?
N1" C6" C5" 122.3(2) . . ?
N1" C6" H6" 118.8 . . ?
C5" C6" H6" 118.8 . . ?
N1 C2 C3 122.1(2) . . ?
N1 C2 C2' 113.98(18) . . ?
C3 C2 C2' 123.9(2) . . ?
C2 C3 C4 118.7(2) . . ?
C2 C3 H3 120.6 . . ?
C4 C3 H3 120.6 . . ?
C5 C4 C3 119.1(2) . . ?
C5 C4 H4 120.4 . . ?
C3 C4 H4 120.4 . . ?
C6 C5 C4 119.4(2) . . ?
C6 C5 H5 120.3 . . ?
C4 C5 H5 120.3 . . ?

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N1 C6 C5 122.0(2) . . ?
N1 C6 H6 119.0 . . ?
C5 C6 H6 119.0 . . ?
C2''' C1''' C6''' 117.9(2) . . ?
C2''' C1''' C4' 121.54(19) . . ?
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C3''' C2''' H2''' 119.5 . . ?
C1''' C2''' H2''' 119.5 . . ?
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C2''' C3''' H3''' 119.5 . . ?
C4''' C3''' H3''' 119.5 . . ?
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C6''' C5''' C4''' 121.4(2) . . ?
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C5''' C6''' H6''' 119.7 . . ?
C1''' C6''' H6''' 119.7 . . ?
O C7 C4''' 109.7(2) . . ?
O C7 H7A 109.7 . . ?
C4''' C7 H7A 109.7 . . ?
O C7 H7B 109.7 . . ?
C4''' C7 H7B 109.7 . . ?
H7A C7 H7B 108.2 . . ?
C7 O C01 116.7(2) . . ?
O C01 C02 108.3(2) . . ?
O C01 H01A 110.0 . . ?
C02 C01 H01A 110.0 . . ?
O C01 H01B 110.0 . . ?
C02 C01 H01B 110.0 . . ?
H01A C01 H01B 108.4 . . ?
N01'' C02 N01 109.6(2) . . ?
N01'' C02 N01' 107.9(2) . . ?
N01 C02 N01' 107.3(2) . . ?
N01'' C02 C01 112.0(2) . . ?
N01 C02 C01 109.8(2) . . ?
N01' C02 C01 110.1(2) . . ?
N6 C9 C8 172.6(18) . . ?

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O51 Cu N1' C6' 88.37(16) . . . . ?
O41 Cu N1' C2' 90.6(2) . . . . ?
N1 Cu N1' C2' -0.02(15) . . . . ?
N1" Cu N1' C2' 178.87(16) . . . . ?
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N1' Cu N1" C6" -179.22(19) . . . . ?
O41 Cu N1" C6" -20.18(19) . . . . ?
N1 Cu N1" C6" -176.1(2) . . . . ?
O51 Cu N1" C6" 62.47(19) . . . . ?
N1' Cu N1" C2" 2.28(14) . . . . ?
O41 Cu N1" C2" 161.32(15) . . . . ?
N1 Cu N1" C2" 5.4(3) . . . . ?
O51 Cu N1" C2" -116.03(15) . . . . ?
N1' Cu N1 C6 -176.6(2) . . . . ?
O41 Cu N1 C6 24.4(2) . . . . ?
N1" Cu N1 C6 -179.7(2) . . . . ?
O51 Cu N1 C6 -58.3(2) . . . . ?
N1' Cu N1 C2 -0.60(15) . . . . ?
O41 Cu N1 C2 -159.60(15) . . . . ?
N1" Cu N1 C2 -3.7(3) . . . . ?
O51 Cu N1 C2 117.68(15) . . . . ?
O42 N4 O41 Cu 171.98(17) . . . . ?
O43 N4 O41 Cu -6.8(2) . . . . ?
N1' Cu O41 N4 13.9(3) . . . . ?
N1 Cu O41 N4 100.97(15) . . . . ?
N1" Cu O41 N4 -70.75(15) . . . . ?
O51 Cu O41 N4 -164.78(15) . . . . ?
O52 N5 O51 Cu 2.0(3) . . . . ?
O53 N5 O51 Cu -178.17(14) . . . . ?
N1' Cu O51 N5 12.00(17) . . . . ?
O41 Cu O51 N5 -168.53(15) . . . . ?
N1 Cu O51 N5 -69.66(15) . . . . ?
N1" Cu O51 N5 92.99(15) . . . . ?
N02 N01 C05 C04 1.0(3) . . . . ?
C02 N01 C05 C04 174.5(3) . . . . ?
N01 C05 C04 C03 -1.0(3) . . . . ?
C05 C04 C03 N02 0.8(4) . . . . ?
C04 C03 N02 N01 -0.2(4) . . . . ?
C05 N01 N02 C03 -0.5(3) . . . . ?
C02 N01 N02 C03 -174.5(2) . . . . ?
N02' N01' C05' C04' 0.0(4) . . . . ?
C02 N01' C05' C04' -177.6(3) . . . . ?
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N01' C05' C04' C03' -0.2(4) . . . . ?
C05' C04' C03' N02' 0.4(4) . . . . ?
C04' C03' N02' N01' -0.4(4) . . . . ?
C05' N01' N02' C03' 0.3(3) . . . . ?
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N02" N01" C05" C04" -0.2(4) . . . . ?
C02 N01" C05" C04" -171.8(3) . . . . ?
N01" C05" C04" C03" 0.4(4) . . . . ?
C05" C04" C03" N02" -0.4(5) . . . . ?
C04" C03" N02" N01" 0.3(4) . . . . ?
C05" N01" N02" C03" 0.0(4) . . . . ?
C02 N01" N02" C03" 172.5(3) . . . . ?
C6' N1' C2' C3' 0.7(3) . . . . ?
Cu N1' C2' C3' 179.90(15) . . . . ?
C6' N1' C2' C2 -178.64(18) . . . . ?
Cu N1' C2' C2 0.6(2) . . . . ?
N1' C2' C3' C4' 0.0(3) . . . . ?
C2 C2' C3' C4' 179.25(19) . . . . ?
C2' C3' C4' C5' -0.8(3) . . . . ?
C2' C3' C4' C1"' 178.21(19) . . . . ?
C3' C4' C5' C6' 0.8(3) . . . . ?
C1"' C4' C5' C6' -178.16(19) . . . . ?
C2' N1' C6' C5' -0.7(3) . . . . ?
Cu N1' C6' C5' -179.86(15) . . . . ?
C2' N1' C6' C2" -179.61(18) . . . . ?
Cu N1' C6' C2" 1.2(2) . . . . ?
C4' C5' C6' N1' -0.1(3) . . . . ?
C4' C5' C6' C2" 178.68(19) . . . . ?
C6" N1" C2" C3" 0.0(3) . . . . ?
Cu N1" C2" C3" 178.65(16) . . . . ?
C6" N1" C2" C6' 179.06(18) . . . . ?
Cu N1" C2" C6' -2.3(2) . . . . ?
N1' C6' C2" N1" 0.8(3) . . . . ?
C5' C6' C2" N1" -178.0(2) . . . . ?
N1' C6' C2" C3" 179.85(19) . . . . ?
C5' C6' C2" C3" 1.0(3) . . . . ?
N1" C2" C3" C4" -0.3(3) . . . . ?
C6' C2" C3" C4" -179.2(2) . . . . ?
C2" C3" C4" C5" 0.2(3) . . . . ?
C3" C4" C5" C6" 0.1(3) . . . . ?
C2" N1" C6" C5" 0.3(3) . . . . ?
Cu N1" C6" C5" -178.11(16) . . . . ?
C4" C5" C6" N1" -0.4(3) . . . . ?
C6 N1 C2 C3 -0.7(3) . . . . ?
Cu N1 C2 C3 -177.01(17) . . . . ?
C6 N1 C2 C2' 177.38(19) . . . . ?
Cu N1 C2 C2' 1.0(2) . . . . ?
N1' C2' C2 N1 -1.1(3) . . . . ?
C3' C2' C2 N1 179.7(2) . . . . ?

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N1' C2' C2 C3 177.0(2) . . . . ?
C3' C2' C2 C3 -2.3(3) . . . . ?
N1 C2 C3 C4 1.4(3) . . . . ?
C2' C2 C3 C4 -176.5(2) . . . . ?
C2 C3 C4 C5 -0.6(3) . . . . ?
C3 C4 C5 C6 -0.8(4) . . . . ?
C2 N1 C6 C5 -0.8(3) . . . . ?
Cu N1 C6 C5 175.00(18) . . . . ?
C4 C5 C6 N1 1.6(4) . . . . ?
C3' C4' C1''' C2''' 160.9(2) . . . . ?
C5' C4' C1''' C2''' -20.2(3) . . . . ?
C3' C4' C1''' C6''' -18.9(3) . . . . ?
C5' C4' C1''' C6''' 160.1(2) . . . . ?
C6''' C1''' C2''' C3''' 0.6(4) . . . . ?
C4' C1''' C2''' C3''' -179.2(2) . . . . ?
C1''' C2''' C3''' C4''' -0.1(4) . . . . ?
C2''' C3''' C4''' C5''' 0.2(4) . . . . ?
C2''' C3''' C4''' C7 -179.5(3) . . . . ?
C3''' C4''' C5''' C6''' -0.8(4) . . . . ?
C7 C4''' C5''' C6''' 178.9(3) . . . . ?
C4''' C5''' C6''' C1''' 1.3(4) . . . . ?
C2''' C1''' C6''' C5''' -1.2(3) . . . . ?
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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C3 C 0.7279(3) 0.4793(2) 0.42969(14) 0.0218(5) Uani 1 1 d . . .
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C4 C 0.8315(3) 0.3857(2) 0.45324(15) 0.0237(5) Uani 1 1 d . . .
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C5 C 0.8765(3) 0.3849(2) 0.52339(15) 0.0226(5) Uani 1 1 d . . .
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C3' C 0.4970(3) 0.6897(2) 0.39361(13) 0.0193(5) Uani 1 1 d . . .
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C1"' C 0.3159(3) 0.8083(2) 0.31222(13) 0.0196(5) Uani 1 1 d . . .
C2"' C 0.1953(3) 0.8986(2) 0.30434(15) 0.0304(6) Uani 1 1 d . . .
H2"' H 0.1598 0.9493 0.3446 0.036 Uiso 1 1 calc R . .
C3"' C 0.1258(3) 0.9168(2) 0.24006(15) 0.0306(6) Uani 1 1 d . . .
H3"' H 0.0438 0.9803 0.2365 0.037 Uiso 1 1 calc R . .
C4"' C 0.1720(3) 0.8456(2) 0.18094(14) 0.0243(5) Uani 1 1 d . . .
C5"' C 0.2918(4) 0.7550(3) 0.18764(16) 0.0408(8) Uani 1 1 d . . .
H5"' H 0.3265 0.7048 0.1470 0.049 Uiso 1 1 calc R . .
C6"' C 0.3623(4) 0.7360(3) 0.25212(16) 0.0401(8) Uani 1 1 d . . .
H6"' H 0.4440 0.6723 0.2555 0.048 Uiso 1 1 calc R . .
C7 C 0.0845(3) 0.8636(3) 0.11395(15) 0.0312(6) Uani 1 1 d . . .
H7A H -0.0221 0.8289 0.1247 0.037 Uiso 1 1 calc R . .
H7B H 0.0657 0.9509 0.1019 0.037 Uiso 1 1 calc R . .
O O 0.1744(2) 0.8088(2) 0.05358(10) 0.0357(5) Uani 1 1 d . . .
C01 C 0.0810(3) 0.8025(3) -0.00483(14) 0.0307(6) Uani 1 1 d . . .
H01A H 0.0334 0.8837 -0.0183 0.037 Uiso 1 1 calc R . .
H01B H -0.0082 0.7493 0.0104 0.037 Uiso 1 1 calc R . .
C02 C 0.1930(3) 0.7522(2) -0.07096(14) 0.0286(6) Uani 1 1 d . . .
N01 N 0.2459(3) 0.6269(2) -0.05671(12) 0.0343(6) Uani 1 1 d . . .
N02 N 0.1782(3) 0.5651(2) 0.00441(13) 0.0362(6) Uani 1 1 d . . .
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C03 C 0.2387(4) 0.4536(3) -0.00443(18) 0.0405(7) Uani 1 1 d . . .
H03 H 0.2143 0.3888 0.0305 0.049 Uiso 1 1 calc R . .
C04 C 0.3413(4) 0.4410(3) -0.07013(18) 0.0501(9) Uani 1 1 d . . .
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C05 C 0.3441(5) 0.5538(3) -0.10240(18) 0.0515(9) Uani 1 1 d . . .
H05 H 0.4041 0.5769 -0.1486 0.062 Uiso 1 1 calc R . .
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C04' C 0.0464(4) 0.7666(3) -0.24886(16) 0.0358(7) Uani 1 1 d . . .
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C05' C 0.1721(4) 0.7602(2) -0.20827(15) 0.0304(6) Uani 1 1 d . . .
H05' H 0.2840 0.7545 -0.2270 0.037 Uiso 1 1 calc R . .
C03" C 0.4521(4) 0.9834(4) -0.10717(19) 0.0493(9) Uani 1 1 d . . .

H03" H 0.4739 1.0644 -0.1215 0.059 Uiso 1 1 calc R . .
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H04" H 0.6710 0.9108 -0.0726 0.069 Uiso 1 1 calc R . .
C05" C 0.4851(4) 0.7964(4) -0.06661(18) 0.0500(9) Uani 1 1 d . . .
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Cl2 Cl 0.57887(8) 0.62474(6) 0.71978(4) 0.03203(16) Uani 1 1 d . . .
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C C 0.9161(5) 0.6069(3) 0.2312(2) 0.0510(9) Uani 1 1 d . . .
C' C 1.0268(5) 0.5347(3) 0.1807(2) 0.0532(9) Uani 1 1 d . . .
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C05 0.067(2) 0.051(2) 0.0263(16) 0.0067(14) 0.0106(15) 0.0249(17)
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C05' 0.0370(15) 0.0313(15) 0.0227(14) -0.0015(10) -0.0045(12) 0.0006(11)
C03" 0.0427(19) 0.065(2) 0.043(2) -0.0165(16) -0.0028(15) -0.0177(16)
C04" 0.0307(18) 0.098(3) 0.048(2) -0.014(2) -0.0075(16) -0.0137(19)
C05" 0.0277(16) 0.083(3) 0.0363(18) 0.0034(16) -0.0069(14) 0.0126(16)
Cl1 0.0199(3) 0.0167(3) 0.0290(3) -0.0040(2) -0.0093(2) 0.0012(2)
Cl2 0.0396(4) 0.0276(3) 0.0244(3) 0.0050(2) 0.0040(3) 0.0078(3)
N 0.116(3) 0.052(2) 0.114(3) -0.036(2) 0.078(3) -0.030(2)
C 0.063(2) 0.0304(18) 0.054(2) -0.0076(14) 0.0236(18) -0.0120(15)
C' 0.067(2) 0.041(2) 0.044(2) -0.0055(15) 0.0152(17) 0.0060(16)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
;

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Zn N1 2.1818(19) . ?
Zn Cl2 2.2499(7) . ?
Zn Cl1 2.2704(7) . ?
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N1 C6 1.335(3) . ?
N1' C2' 1.334(3) . ?
N1' C6' 1.339(3) . ?
N1" C6" 1.337(3) . ?

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C3 C4 1.388(3) . ?
C3 H3 0.9500 . ?
C4 C5 1.372(4) . ?
C4 H4 0.9500 . ?
C5 C6 1.380(3) . ?
C5 H5 0.9500 . ?
C6 H6 0.9500 . ?
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C3' C4' 1.394(3) . ?
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C3" H3" 0.9500 . ?
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C4" H4" 0.9500 . ?
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C7 H7B 0.9900 . ?
O C01 1.403(3) . ?
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C02 N01' 1.453(3) . ?
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N01 N02 1.353(3) . ?

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N01' N02' 1.353(3) . ?
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N01" N02" 1.359(4) . ?
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C03 C04 1.379(4) . ?
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C04 C05 1.358(5) . ?
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N1" Zn Cl1 97.90(5) . . ?
N1 Zn Cl1 98.99(5) . . ?
Cl2 Zn Cl1 119.75(3) . . ?
C2 N1 C6 119.3(2) . . ?
C2 N1 Zn 115.65(15) . . ?
C6 N1 Zn 124.95(17) . . ?
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C2 C3 C4 118.2(2) . . ?
C2 C3 H3 120.9 . . ?
C4 C3 H3 120.9 . . ?
C5 C4 C3 119.5(2) . . ?
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N1' C2' C3' 121.7(2) . . ?
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C3' C2' C2 124.7(2) . . ?
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N1' C6' C5' 121.6(2) . . ?
N1' C6' C2" 113.9(2) . . ?
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O C7 C4"' 110.3(2) . . ?
O C7 H7A 109.6 . . ?
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C4"' C7 H7B 109.6 . . ?
H7A C7 H7B 108.1 . . ?
C7 O C01 111.0(2) . . ?
O C01 C02 107.4(2) . . ?
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N01' C02 C01 108.8(2) . . ?
C05 N01 N02 111.4(2) . . ?
C05 N01 C02 127.9(2) . . ?
N02 N01 C02 120.1(2) . . ?
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C05' N01' C02 127.6(2) . . ?
N02' N01' C02 119.8(2) . . ?
C03' N02' N01' 103.8(2) . . ?
C05" N01" N02" 111.0(3) . . ?
C05" N01" C02 129.5(3) . . ?
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C04" C05" H05" 126.6 . . ?
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C C' H'1 109.5 . . ?
C C' H'2 109.5 . . ?
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N1' Zn N1" C6" 178.6(2) . . . . ?
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Cl1 Zn N1" C6" -75.28(19) . . . . ?
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C6 N1 C2 C3 -0.3(3) . . . . ?
Zn N1 C2 C3 176.11(17) . . . . ?
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Zn N1 C2 C2' -6.0(2) . . . . ?
N1 C2 C3 C4 -0.2(3) . . . . ?
C2' C2 C3 C4 -177.9(2) . . . . ?
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Zn N1 C6 C5 -175.66(16) . . . . ?
C4 C5 C6 N1 0.1(3) . . . . ?
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Zn N1' C2' C3' -173.61(16) . . . . ?
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C3 C2 C2' N1' 177.2(2) . . . . ?
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C2' N1' C6' C2" 178.91(19) . . . . ?
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C02 N01 N02 C03 172.8(3) . . . . ?
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N01 C02 N01' N02' 98.9(3) . . . . ?
C01 C02 N01' N02' -23.4(3) . . . . ?
C05' N01' N02' C03' -0.6(3) . . . . ?
C02 N01' N02' C03' -176.7(2) . . . . ?
N01 C02 N01" C05" -24.6(4) . . . . ?
N01' C02 N01" C05" -143.5(3) . . . . ?
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N01 C02 N01" N02" 172.3(2) . . . . ?
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C02 N01" N02" C03" 168.3(2) . . . . ?
N01 N02 C03 C04 -1.0(4) . . . . ?
N02 C03 C04 C05 0.9(4) . . . . ?
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;
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University of Canterbury,
Private Bag 4800,
Christchurch,
New Zealand
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bis(2,4-bis(2-pyridyl-\kN)-6-(4-pyridyl)-1,3,5-triazine-\kN^3^)ruthenium(II)
bis(hexafluorophosphate)
;

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bis(hexafluorophosphate)
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Crystals of the title compound, [Ru(C~18~H~12~N~6~)~2~](PF~6~)~2~,
 are orthorhombic, R = 0.0584; Rw(F2) = 0.1295 for 2935 reflections.
 The coordination sphere is formed by six N atoms from the tridentate sites of
 two ligands that are meridionally oriented around the Ru(II) ion. The locations
 of the donor atoms deviate substantially from an ideal octahedron. The Ru-N
 distances are in the range 1.983(4) -- 2.111(4) \%A. The Ru atom occupies a
 special position with twofold rotational symmetry. One of the P atoms occupies
 a special position with twofold rotational symmetry and the other P atom is
 located on a special position at a centre of symmetry.
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_publ_section_comment
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Our aim involves the synthesis and characterisation of bridging ligands
 containing two non-equivalent binding sites. Our interests are particularly
 focused on preparation of such ligands capable of binding a Ru(II) ion at one
 site and a Co(III) ion at the other site. As part of our study, the ligand
 2,4-bis(2-pyridyl)-6-(4-pyridyl)-1,3,5-triazine was synthesised using
 the method of Polson et al. (2002). This terpyridyl-like ligand, which
 contains a remote fourth nitrogen donor atom, was a candidate for our
 study. The ruthenium complex (I) in which two ligands are coordinated to a
 Ru(II) ion at their tridentate sites was prepared according to the reported
 methods (Polson et al. (2002 and 2004)).

Complex (I) (Fig. 1) crystallises in the orthorhombic space group Pcca with
 four well separated molecules in the unit cell. The asymmetric unit comprises
 half a molecule of (I) and two halves of non-coordinated hexafluorophosphate
 anions. The ruthenium ion in compound (I) is six-coordinate, but the geometry
 is significantly distorted from that of an ideal octahedron as a result of the
 constraints enforced by the ligand structure. This is commonly observed for
 terpyridyl-like ligands (Thummel & Jahng, 1986; Constable et al., 1990, 1992,
 2001; Cathey et al., 1990; Bushell et al., 1998; Craig et al., 1998;
 Sasaki et al., 1998; Cardenas et al. 1999; Chamchoumis & Potvin, 1999;
 Fallahpour et al. 1999; Lashgari et al., 1999; Pyo et al., 1999; Ziegler
 et al., 1999; Alcock et al., 2000; Encinas et al., 2002; Fang et al., 2002;
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Laine et al., 2002). Each of the tridentate ligands occupies
three meridionally located coordination sites. The ruthenium-nitrogen bond
lengths show the expected pattern for complexes related to
[Ru(terpy)~2~](PF~6~)~2~ (Pyo et al., 1999;
Lashgari et al., 1999), with short metal-ligand bonds to the central ring
(1.983(4) \%A) and longer bonds to the side pyridine rings (mean; 2.108(4)
\%A). The non-coordinated pyridine rings are not coplanar with the central
triazine ring and form interannular angles of 16.8(0.30)\%. Similar
observations have been made for the Ni(II) and Ru(II) complexes based on the
4'-phenyl substituted terpyridine system (Constable et al., 1990;
Chamchoumis & Potvin, 1999). As shown in Fig. 2, there are \p-\p stacking
interactions (face-face) between the terminal pyridyl rings of the adjacent
complexes (centroid-centroid 3.72(8) \%A). The complex cations are arranged
like the steps of a spiral staircase along the b-axis of the unit cell,
with the stack of pyridyl rings forming the pole of the staircase.
There appears to be no significant hydrogen bonding within the lattice.
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The 2,4-bis(2-pyridyl)-6-(4-pyridyl)-1,3,5-triazine ligand and its
ruthenium complex (I) were synthesised according to the methods of Polson
et al. (2002 and 2004). Red block crystals were obtained by slow evaporation
of CH~3~CN-H~2~O solution of the complex (yield 16%).
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All H atoms were placed in calculated positions [C-H = 0.93 \%A and U~iso~ =
1.2 U~eq~ (C)] and were included in the refinement in the riding-model
approximation.
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_publ_section_references
;
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Dupic, G.; Daran, J.-C.; Balavoine, G. G. A. (1999). J. Chem. Soc., Dalton
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Table 1. Selected geometric parameters (\%A, \%).

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Fig. 1. A perspective view of the Ru(II) complex (I), showing the
atom-labelling scheme with 50% probability thermal displacement
ellipsoids. Unlabelled atoms in the cation, and the PF~6~^-^ anions
(P~1~ and P~2~) are related by the symmetry codes (1/2-x, -y, z),
(1/2-x, 1-y, z), and (1-x, 2-y, -z), respectively. H atoms are drawn
as small spheres of arbitrary radii.

Fig. 2. A packing diagram, showing the \p-\p stacking interactions between
the complexes parallel to the ac plane.
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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C16 0.060(4) 0.028(3) 0.080(5) -0.006(3) 0.018(4) -0.010(3)
C17 0.058(4) 0.050(4) 0.073(5) -0.010(4) 0.012(4) -0.016(4)
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F21 0.0401(18) 0.0398(18) 0.043(2) -0.0144(15) -0.0049(15) 0.0077(14)
F22 0.0435(19) 0.0370(18) 0.051(2) 0.0037(16) 0.0091(16) 0.0034(15)
F23 0.0381(17) 0.0349(17) 0.0372(18) -0.0047(14) -0.0001(14) 0.0099(14)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
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 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
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 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
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C8 C 0.5243(3) 0.1577(2) 0.82255(14) 0.0255(7) Uani 1 1 d . . .
H8 H 0.5588 0.1620 0.7852 0.031 Uiso 1 1 calc R . .
C9 C 0.1390(3) 0.42119(19) 0.80606(13) 0.0210(6) Uani 1 1 d . . .
C10 C 0.0533(3) 0.4854(2) 0.80282(14) 0.0275(7) Uani 1 1 d . . .
H10 H 0.0067 0.4988 0.8357 0.033 Uiso 1 1 calc R . .
C11 C 0.0369(3) 0.5298(2) 0.75048(15) 0.0332(8) Uani 1 1 d . . .
H11 H -0.0200 0.5752 0.7473 0.040 Uiso 1 1 calc R . .
C12 C 0.1041(3) 0.5072(2) 0.70289(15) 0.0343(8) Uani 1 1 d . . .
H12 H 0.0928 0.5363 0.6665 0.041 Uiso 1 1 calc R . .
C13 C 0.1879(3) 0.4418(2) 0.70911(14) 0.0270(7) Uani 1 1 d . . .
H13 H 0.2335 0.4262 0.6764 0.032 Uiso 1 1 calc R . .
C14 C 0.0629(3) 0.3222(2) 1.00480(13) 0.0264(7) Uani 1 1 d . . .
C15 C 0.0870(3) 0.2609(2) 1.04568(14) 0.0300(7) Uani 1 1 d . . .
H15 H 0.1448 0.2175 1.0389 0.036 Uiso 1 1 calc R . .
C16 C 0.0262(4) 0.2631(3) 1.09661(16) 0.0400(9) Uani 1 1 d . . .
H16 H 0.0416 0.2212 1.1259 0.048 Uiso 1 1 calc R . .
C17 C -0.0576(3) 0.3263(3) 1.10537(15) 0.0370(9) Uani 1 1 d . . .
H17 H -0.1008 0.3283 1.1405 0.044 Uiso 1 1 calc R . .
C18 C -0.0778(3) 0.3866(3) 1.06260(16) 0.0399(9) Uani 1 1 d . . .
H18 H -0.1351 0.4305 1.0689 0.048 Uiso 1 1 calc R . .
Cl1 Cl 0.18457(7) 0.20129(5) 0.74884(3) 0.02395(17) Uani 1 1 d . . .
Cl2 Cl 0.48121(8) 0.41149(6) 0.81084(4) 0.0339(2) Uani 1 1 d . . .
Fe' Fe 0.50388(4) 0.28090(3) 0.642323(19) 0.02480(13) Uani 1 1 d . . .
N1' N 0.5982(3) 0.26540(17) 0.56352(11) 0.0261(6) Uani 1 1 d . . .
N2' N 0.6268(3) 0.19034(18) 0.47619(11) 0.0287(6) Uani 1 1 d . . .
N3' N 0.7535(3) 0.31014(19) 0.50210(11) 0.0298(6) Uani 1 1 d . . .
N4' N 0.6395(2) 0.38263(17) 0.64003(12) 0.0266(6) Uani 1 1 d . . .
N5' N 0.4217(2) 0.17166(17) 0.59599(11) 0.0253(6) Uani 1 1 d . . .
N6' N 0.8774(3) 0.2873(2) 0.40244(13) 0.0383(8) Uani 1 1 d . . .
C1' C 0.5687(3) 0.2039(2) 0.52559(13) 0.0257(7) Uani 1 1 d . . .
C2' C 0.7187(3) 0.2458(2) 0.46652(14) 0.0290(7) Uani 1 1 d . . .
C3' C 0.6889(3) 0.3173(2) 0.55010(14) 0.0273(7) Uani 1 1 d . . .
C4' C 0.7116(3) 0.3857(2) 0.59335(14) 0.0283(7) Uani 1 1 d . . .
C5' C 0.7965(3) 0.4492(2) 0.58617(16) 0.0344(8) Uani 1 1 d . . .
H5' H 0.8465 0.4496 0.5532 0.041 Uiso 1 1 calc R . .
C6' C 0.8068(3) 0.5124(2) 0.62819(17) 0.0383(9) Uani 1 1 d . . .
H6' H 0.8633 0.5574 0.6241 0.046 Uiso 1 1 calc R . .
C7' C 0.7337(3) 0.5091(2) 0.67626(16) 0.0335(8) Uani 1 1 d . . .
H7' H 0.7401 0.5515 0.7058 0.040 Uiso 1 1 calc R . .
C8' C 0.6513(3) 0.4432(2) 0.68045(15) 0.0293(7) Uani 1 1 d . . .

H8' H 0.6013 0.4411 0.7134 0.035 Uiso 1 1 calc R . .
C9' C 0.4655(3) 0.1510(2) 0.54286(14) 0.0260(7) Uani 1 1 d . . .
C10' C 0.4159(3) 0.0861(2) 0.50849(14) 0.0282(7) Uani 1 1 d . . .
H10' H 0.4461 0.0744 0.4709 0.034 Uiso 1 1 calc R . .
C11' C 0.3217(3) 0.0386(2) 0.52990(16) 0.0335(8) Uani 1 1 d . . .
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C12' C 0.2793(3) 0.0582(2) 0.58455(16) 0.0345(8) Uani 1 1 d . . .
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C14' C 0.7826(3) 0.2351(2) 0.41099(14) 0.0324(8) Uani 1 1 d . . .
C15' C 0.7409(4) 0.1765(3) 0.36954(16) 0.0430(10) Uani 1 1 d . . .
H15' H 0.6750 0.1395 0.3774 0.052 Uiso 1 1 calc R . .
C16' C 0.7969(4) 0.1726(3) 0.31638(17) 0.0495(11) Uani 1 1 d . . .
H16' H 0.7686 0.1339 0.2868 0.059 Uiso 1 1 calc R . .
C17' C 0.8933(4) 0.2250(3) 0.30700(16) 0.0440(10) Uani 1 1 d . . .
H17' H 0.9333 0.2233 0.2709 0.053 Uiso 1 1 calc R . .
C18' C 0.9319(4) 0.2808(3) 0.35100(16) 0.0417(10) Uani 1 1 d . . .
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Cl1' Cl 0.66678(8) 0.19572(5) 0.68666(4) 0.03177(19) Uani 1 1 d . . .
Cl2' Cl 0.37069(8) 0.37540(5) 0.58502(3) 0.0315(2) Uani 1 1 d . . .
O O 0.4214(2) 0.28892(14) 0.70697(9) 0.0267(5) Uani 1 1 d . . .
O1 O 0.3336(2) 0.52964(15) 0.89297(10) 0.0330(5) Uani 1 1 d . . .
H1A H 0.3301 0.5853 0.8678 0.050 Uiso 1 1 d R . .
H1B H 0.3820 0.4855 0.8687 0.050 Uiso 1 1 d R . .
O2' O 0.1078(8) 0.4519(10) 0.5617(4) 0.076(6) Uani 0.294(9) 1 d P . .
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C4 0.0222(16) 0.0205(15) 0.0211(14) -0.0040(11) 0.0011(12) -0.0011(13)
C5 0.0298(18) 0.0271(17) 0.0202(15) -0.0015(12) 0.0004(12) 0.0001(14)
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C8 0.0244(17) 0.0268(17) 0.0261(16) -0.0039(13) 0.0066(13) 0.0024(14)
C9 0.0188(16) 0.0203(15) 0.0241(15) -0.0027(12) 0.0033(12) -0.0020(12)
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C13 0.0280(18) 0.0261(17) 0.0275(16) 0.0029(13) 0.0077(13) 0.0034(14)
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Cl2 0.0271(4) 0.0330(5) 0.0420(5) -0.0115(4) 0.0067(3) -0.0050(3)
Fe' 0.0281(3) 0.0249(3) 0.0224(2) -0.00621(17) 0.00974(18) -0.0057(2)
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N6' 0.0411(19) 0.0424(19) 0.0327(16) 0.0087(13) 0.0149(13) 0.0122(15)
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C8' 0.0281(18) 0.0308(18) 0.0291(17) -0.0026(13) 0.0030(13) 0.0005(15)
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C10' 0.0269(18) 0.0292(18) 0.0278(16) -0.0093(13) -0.0031(13) 0.0095(14)
C11' 0.0295(19) 0.0277(18) 0.042(2) -0.0135(15) -0.0060(15) 0.0060(15)
C12' 0.0270(19) 0.0301(19) 0.047(2) -0.0040(15) 0.0053(15) -0.0026(15)
C13' 0.0273(18) 0.0304(18) 0.0302(17) -0.0054(14) 0.0051(13) 0.0003(14)
C14' 0.0287(19) 0.045(2) 0.0238(16) 0.0057(14) 0.0051(13) 0.0153(16)
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C16' 0.039(2) 0.081(3) 0.0289(19) -0.0099(19) 0.0069(16) 0.006(2)
C17' 0.040(2) 0.068(3) 0.0254(18) 0.0074(17) 0.0116(15) 0.022(2)
C18' 0.045(2) 0.048(2) 0.0346(19) 0.0146(17) 0.0175(17) 0.0161(19)
Cl1' 0.0298(4) 0.0366(5) 0.0297(4) -0.0041(3) 0.0080(3) 0.0021(4)
Cl2' 0.0393(5) 0.0310(4) 0.0246(4) -0.0023(3) 0.0062(3) -0.0006(4)
O 0.0280(12) 0.0267(12) 0.0262(11) -0.0015(9) 0.0093(9) -0.0028(10)
O1 0.0354(14) 0.0341(14) 0.0303(12) 0.0006(10) 0.0077(10) -0.0019(11)
O2' 0.022(5) 0.177(15) 0.028(5) -0.014(6) -0.007(4) 0.033(7)
O2 0.215(9) 0.162(8) 0.123(6) -0.079(5) 0.141(6) -0.142(7)

_geom_special_details
;
All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
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Fe Cl1 2.4187(9) . ?
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Hexaaquacopper(II) dinitrate -- absence of Jahn-Teller distortion
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Hexaaquacopper(II) dinitrate
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Cu H12 O6, 2(N O3)
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_publ_section_abstract
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In the title compound, [Cu(H~2~O)~6~](NO~3~)~2~,
the geometry around the Cu(II) ion is approximately
an octahedron. The coordination sphere is formed
by six O atoms from the coordinated water molecules
around the Cu(II) ion. The Cu-O distances are rather
similar (2.014(2) -- 2.084(2) \%A) and not related by
symmetry. The Jahn-Teller effect is, at best, only
weakly observed in this structure, in contrast to
many other structures where the hexaaquacopper(II)
ion has been characterised. An extensive mesh of
hydrogen bond interactions between the coordinated
water molecules and nitrate ions is a feature of the
structure and may be limiting the degree to which
the Jahn-Teller effect can be observed.

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Blue block-like crystals were obtained from attempts
to prepare and isolate the copper complex of the
ditopic ligand 1-[4'-p-tolyl-(2,2':6',2"-terpyridyl)
-1,4,8,11-tetraazacyclotetradecane (L), (I) (Padilla-Tosta
et al., 2000). An X-ray diffraction study of
these crystals revealed that hexaaquacopper(II)
dinitrate, (II), had crystallised from the mixture.
Attempts to grow similar crystals in the absence of
the ditopic ligand proved unsuccessful, which leads
us to speculate that the ditopic ligand may be
influencing the crystallisation process.
Unfortunately, the vagaries of nucleation and crystal
growth make it difficult to test this hypothesis.
Here, we report the structure of hexaaquacopper
complex as its dinitrate salt (II).

The copper complex (II) (Fig. 1) crystallises in the
triclinic space group P-1 with two molecules in the
unit cell. The asymmetric unit consists of a
[Cu(H~2~O)~6~]^2+^ cation and two nitrate anions.

The geometry around the Cu^2+^ can be best described as an octahedron, with bonds to six water molecules (Fig. 1 and Table 1). The Cu-O bond distances are rather similar, falling in the range 2.014(2) -- 2.084(2) \%A, and there is an extended hydrogen bonding network that links the coordinated water molecules and the nitrate anions throughout the crystal lattice (Fig. 2).

The similarity of the copper-oxygen bond lengths is relatively unusual in that Jahn-Teller distortion often leads to two of the copper-ligand bonds that lie along one axis being much longer than the remaining four copper-ligand bonds. A number of Jahn-Teller distorted hexaaquacopper complexes have been characterised by X-ray crystallography (Averbuch-Pouchot & Durif, 1989; Benedetti et al., 1979, 1986; Bernardinelli et al., 1991; Couldwell et al., 1978; Dalrymple et al., 2002; Durif & Averbuch-Pouchot; Filippova, 2000; Glowiak & Podgorska, 1986; 1987; Honghui et al., 1988; Kennard & Smith, 1989; Li et al., 2004; Ma et al., 2001, 2003; Maslen et al., 1988; Navarro et al., 2000; Rodriguez-Martin et al., 2002; Shamuratov et al., 1993; Wang et al., 1988; Zviedre et al., 1985). In these cases, the axial copper-oxygen bond lengths fall in the range (2.202 -- 2.423 \%A) in comparison to the equatorial bond lengths (1.945 -- 2.084 \%A). The mean axial bond length is between 8.7 and 24% longer than the mean equatorial bond length in these structures (the mean value of these percentage differences is 18.6% over 20 structures). In our structure the mean bond length along the longest axis (O2-Cu-O4) is only 1.6% longer than that along the remaining axes.

We are aware of only six crystallographic studies of copper(II) complexes where static Jahn-Teller distortions are not observed in complexes where all six donors are otherwise identical (Blackburn et al., 1991; Cullen et al., 1970, 1971; Joesten et al., 1970; Ray et al., 1973; Takagi et al., 1976). The structure we report further stands out from these other six, because in this case the copper atom lies on a general position, with all copper-oxygen bonds lengths being independently refined. In the remaining six cases, the copper atoms are located on the special positions in higher symmetry space groups (Pa-3, P-31c, Fm3, P-3c1, R-3, and Fm3, respectively).

Jahn-Teller distortion may not be observed in a crystallographic study if either there is disorder in the structure (so that a defined long axis is randomly distributed over the three

orientations relation to the unit cell axes), or there is
sufficient thermal motion to allow the long and short bonds
in a structure to exchange over time (sometimes referred to
as the dynamic Jahn-Teller effect). In these cases, the
averaging inherent in the X-ray experiment (over spatial
location in the crystal in the first case or time in the
second) might be expected to manifest itself in the
crystallographic modelling process as larger than expected
anisotropic thermal parameters for the donor atoms along the
direction of the copper-ligand bond. This effect has been
discussed (Cullen et al., 1970) and may be significant in a
number of the literature cases (Blackburn et al., 1991;
Cullen et al., 1971; Takagi et al., 1976).

Table 3 presents the principal-axis values for the anisotropic
displacement ellipsoids of the water-O atoms in the structure
(II). The largest principal axes of the ellipsoids are not
directed along the Cu--O bonds (Fig. 4). Taken together,
these data strongly suggest the lack of Jahn-Teller
distortion (static or dynamic) in the structure (II).

In the Complex (II), three marginally longer Cu--O bonds
(Cu--O2, Cu--O3, and Cu--O4) are meridionally distributed
around the copper atom, as are the Cu--O shorter bonds. The
variation in the Cu--O bond lengths of the structure, and the
absence of any significant Jahn-Teller effect may be explained
by the influence of the hydrogen-bonding network in the lattice
of the complex (Fig. 3 and Table 2). All of the coordinated water
molecules are involved in several hydrogen bonds, which means
that while the copper centre may not be in its lowest energy
Jahn-Teller distorted state, this could be made up for by the
large number of weak interactions that may each be marginally
stronger in the less distorted structure.

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_publ_section_acknowledgements
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The authors would like to thank Professor W. T. Robinson and
Dr J. Wikaira for their help with X-ray structural investigations
associated with this project. A University of Canterbury Doctoral
Scholarship (R.Z.) is gratefully acknowledged.
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_publ_section_exptl_prep
;
A solution of Cu(NO~3~)~2~.3H~2~O (50 mg) in ethanol (5 cm^3^)
was added to a cooled, filtered solution of ligand L , I,

(0.15 g) in ethanol (5 cm^3^). The reaction mixture was
heated at reflux for 1 h, and upon cooling to room temperature afforded
a blue-green insoluble precipitate (0.11 g). The precipitate was
suspended in ethanol-water (1:1, 5 cm^3^), then the mixture was
filtered after it was heated to reflux for 1 h. The solution was
allowed to cool to room temperature overnight. The solution was
kept in the refrigerator for about two months during which time
blue crystals of [Cu(OH~2~)~6~](NO~3~)~2~ suitable for X-ray analysis
were produced. No crystals of [Cu(L)](NO~3~)~2~ were
produced in this way.

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_publ_section_exptl_refinement
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All H atoms were placed in calculated positions [C-H = 0.93 \%A and U~iso~ =
1.2 U~eq~ (C)] and were included in the refinement in the riding-model
approximation.
;

_publ_section_references
;
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Table 1. Selected geometric parameters (\%A, \%).

Table 2. Geometric parameters for hydrogen-bonding interactions (\%A, \%).

Table 3. Selected anisotropic displacement parameters (\%A^2^).
;

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Fig. 1. A perspective view of diagram of the Cu(II)
complex, (II), showing the atom-labelling scheme with 50% probability
displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

Fig. 2. A packing diagram, showing hydrogen-bonding interactions within the
lattice of the complex, (II).

Fig. 3. A perspective view of diagram of the Cu(II)
complex, (II), showing the hydrogen-bonding interactions involving the
dication.

Fig. 4. A view of the dication, showing the largest principal axes of the
ellipsoids of the water-O atoms are not directed along the Cu--O bonds.
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'Cu' 0.0058(3)  0.0106(3)  0.0126(3)  0.00344(18) 0.00374(17) 0.00079(17)
'O1' 0.0078(12) 0.0084(13) 0.0119(12) 0.0020(10)  0.0045(10)  0.0012(9)
'O2' 0.0050(11) 0.0102(13) 0.0130(12) 0.0047(10)  0.0024(9)   0.0003(9)
'O3' 0.0091(12) 0.0099(13) 0.0123(12) 0.0032(10)  0.0053(10)  0.0027(10)
'O4' 0.0052(11) 0.0079(13) 0.0134(12) 0.0031(10)  0.0035(9)   0.0013(9)
'O5' 0.0067(12) 0.0120(13) 0.0160(13) 0.0076(10)  0.0064(10)  0.0021(10)
'O6' 0.0058(12) 0.0114(13) 0.0137(12) 0.0063(10)  0.0046(10)  0.0014(9)

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Table 3. Selected anisotropic displacement parameters (\%A^2^).
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 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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 'x, y, z'
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_cell_angle_alpha                 106.428(2)
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
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 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
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 factors based on ALL data will be even larger.
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H1A H 0.230(7) -0.058(5) 0.018(2) 0.011 Uiso 1 1 d D . .
H1B H 0.180(7) -0.189(4) 0.106(3) 0.011 Uiso 1 1 d D . .
O2 O 0.5425(4) 0.2400(4) 0.3146(2) 0.0094(5) Uani 1 1 d D . .
H2A H 0.607(7) 0.372(3) 0.323(3) 0.011 Uiso 1 1 d D . .
H2B H 0.581(7) 0.216(5) 0.394(2) 0.011 Uiso 1 1 d D . .
O3 O 0.0978(4) 0.3582(4) 0.3683(2) 0.0102(5) Uani 1 1 d D . .
H3A H 0.185(7) 0.479(4) 0.365(3) 0.012 Uiso 1 1 d D . .
H3B H 0.138(7) 0.343(5) 0.449(2) 0.012 Uiso 1 1 d D . .
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H4B H -0.234(7) -0.097(3) 0.137(4) 0.011 Uiso 1 1 d D . .
O5 O 0.2220(4) 0.3095(4) 0.1238(2) 0.0105(5) Uani 1 1 d D . .
H5A H 0.388(4) 0.340(5) 0.111(4) 0.013 Uiso 1 1 d D . .
H5B H 0.144(6) 0.412(4) 0.134(4) 0.013 Uiso 1 1 d D . .
O6 O 0.1346(4) -0.0050(4) 0.3531(2) 0.0096(5) Uani 1 1 d D . .
H6A H -0.037(4) -0.048(5) 0.352(4) 0.012 Uiso 1 1 d D . .
H6B H 0.221(6) -0.102(5) 0.356(4) 0.012 Uiso 1 1 d D . .
N1 N 0.7929(5) 0.5522(4) 0.1275(3) 0.0093(6) Uani 1 1 d . . .
O11 O 0.6585(4) 0.6598(4) 0.1232(2) 0.0119(5) Uani 1 1 d . . .
O12 O 0.7080(4) 0.3869(4) 0.1234(2) 0.0124(5) Uani 1 1 d . . .
O13 O 1.0202(4) 0.6086(4) 0.1371(2) 0.0132(6) Uani 1 1 d . . .
N2 N 0.5813(5) 0.7634(4) 0.3903(3) 0.0102(6) Uani 1 1 d . . .
O21 O 0.6533(5) 0.9126(4) 0.3697(2) 0.0124(5) Uani 1 1 d . . .
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Cu 0.0058(3) 0.0106(3) 0.0126(3) 0.00344(18) 0.00374(17) 0.00079(17)
O1 0.0078(12) 0.0084(13) 0.0119(12) 0.0020(10) 0.0045(10) 0.0012(9)
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O2 0.0050(11) 0.0102(13) 0.0130(12) 0.0047(10) 0.0024(9) 0.0003(9)
O3 0.0091(12) 0.0099(13) 0.0123(12) 0.0032(10) 0.0053(10) 0.0027(10)
O4 0.0052(11) 0.0079(13) 0.0134(12) 0.0031(10) 0.0035(9) 0.0013(9)
O5 0.0067(12) 0.0120(13) 0.0160(13) 0.0076(10) 0.0064(10) 0.0021(10)
O6 0.0058(12) 0.0114(13) 0.0137(12) 0.0063(10) 0.0046(10) 0.0014(9)
N1 0.0081(14) 0.0111(17) 0.0077(14) 0.0015(11) 0.0033(11) 0.0011(12)
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O12 0.0095(12) 0.0079(14) 0.0216(14) 0.0068(10) 0.0067(10) 0.0007(10)
O13 0.0051(12) 0.0120(14) 0.0223(14) 0.0058(11) 0.0054(10) -0.0007(10)
N2 0.0080(14) 0.0121(16) 0.0088(14) 0.0009(12) 0.0032(11) 0.0016(12)
O21 0.0119(12) 0.0108(14) 0.0180(13) 0.0078(10) 0.0081(10) 0.0023(10)
O22 0.0065(12) 0.0137(15) 0.0227(14) 0.0049(11) 0.0065(10) -0.0003(10)
O23 0.0106(12) 0.0135(14) 0.0143(13) 0.0037(10) 0.0043(10) 0.0054(10)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
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 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Cu O1 2.034(2) . y
Cu O6 2.041(2) . y
Cu O4 2.064(2) . y
Cu O3 2.074(2) . y
Cu O2 2.084(2) . y
O1 H1A 0.981(18) . ?
O1 H1B 0.966(18) . ?
O2 H2A 0.976(18) . ?
O2 H2B 0.981(18) . ?
O3 H3A 0.977(18) . ?
O3 H3B 0.966(18) . ?
O4 H4A 0.970(18) . ?
O4 H4B 0.974(18) . ?
O5 H5A 0.974(18) . ?
O5 H5B 0.963(18) . ?
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O6 H6B 0.978(18) . ?
N1 O11 1.241(4) . ?

```
N1 O12 1.245(4) . ?
N1 O13 1.268(4) . ?
N2 O21 1.233(4) . ?
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N2 O22 1.272(4) . ?

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O5 Cu O6 175.94(10) . . y
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O5 Cu O4 91.38(10) . . y
O1 Cu O4 88.81(10) . . y
O6 Cu O4 91.34(10) . . y
O5 Cu O3 91.72(10) . . y
O1 Cu O3 178.31(10) . . y
O6 Cu O3 85.17(10) . . y
O4 Cu O3 92.35(10) . . y
O5 Cu O2 89.50(10) . . y
O1 Cu O2 87.93(10) . . y
O6 Cu O2 87.96(10) . . y
O4 Cu O2 176.61(9) . . y
O3 Cu O2 90.89(10) . . y
Cu O1 H1A 116(2) . . ?
Cu O1 H1B 113(2) . . ?
H1A O1 H1B 113(2) . . ?
Cu O2 H2A 112(2) . . ?
Cu O2 H2B 113(2) . . ?
H2A O2 H2B 112(2) . . ?
Cu O3 H3A 108(2) . . ?
Cu O3 H3B 110(2) . . ?
H3A O3 H3B 113(2) . . ?
Cu O4 H4A 111(2) . . ?
Cu O4 H4B 108(2) . . ?
H4A O4 H4B 113(2) . . ?
Cu O5 H5A 114(2) . . ?
Cu O5 H5B 118(2) . . ?
H5A O5 H5B 114(3) . . ?
Cu O6 H6A 110(2) . . ?
Cu O6 H6B 122(2) . . ?
H6A O6 H6B 112(2) . . ?
O11 N1 O12 120.8(3) . . ?
O11 N1 O13 120.7(3) . . ?
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O12 N1 O13 118.5(3) . . ?
O21 N2 O23 121.1(3) . . ?
O21 N2 O22 118.9(3) . . ?
O23 N2 O22 120.0(3) . . ?

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 _geom_hbond_distance_DA
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O1 H1B O13  0.966(18) 1.788(19) 2.741(4) 168(4) 1_445
O2 H2B O22  0.981(18) 2.12(2) 3.038(4) 156(3) 2_666
O2 H2A O23  0.976(18) 2.00(2) 2.940(4) 162(3) .
O2 H2B O21  0.981(18) 2.38(3) 2.912(4) 113(3) 1_545
O3 H3A O22  0.977(18) 1.83(2) 2.779(4) 162(3) .
O3 H3B O23  0.966(18) 1.88(2) 2.827(4) 167(3) 2_666
O4 H4A O2  0.970(18) 1.99(2) 2.942(4) 167(4) 1_455
O4 H4A O1  0.970(18) 2.60(4) 3.070(3) 110(3) 1_455
O4 H4B O11  0.974(18) 1.79(2) 2.763(4) 175(3) 1_445
O5 H5A O12  0.974(18) 1.78(2) 2.735(3) 166(4) .
O5 H5A N1  0.974(18) 2.50(3) 3.417(4) 156(3) .
O5 H5A O11  0.974(18) 2.58(3) 3.285(3) 130(3) .
O5 H5B O13  0.963(18) 1.78(2) 2.740(4) 172(4) 1_455
O5 H5B N1  0.963(18) 2.47(2) 3.365(4) 155(3) 1_455
O5 H5B O12  0.963(18) 2.45(3) 3.123(3) 126(3) 1_455
O6 H6B O21  0.978(18) 2.44(3) 3.154(3) 130(3) 1_545
O6 H6B O22  0.978(18) 1.91(2) 2.860(4) 162(4) 1_545
O6 H6B N2  0.978(18) 2.51(2) 3.436(4) 157(3) 1_545

_diffrn_measured_fraction_theta_max    0.953
_diffrn_reflns_theta_full              25.00
_diffrn_measured_fraction_theta_full   0.953
_refine_diff_density_max    0.598
_refine_diff_density_min   -1.142
_refine_diff_density_rms    0.120
_chemical_compound_source        ?
_exptl_crystal_recrystallization_method
'Slow evaporation from EtOH-water solution of the complex'
#End of CIF============================================================
```

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Appendix 4

Structure Factor Tables

==========

```
#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_[Ni(tpt)(H2O)3]Cl2.3H2O, 2.7 (A)
_shelx_title ' 2.7 (A) in C2/c'
_shelx_refln_list_code          4
_shelx_F_calc_maximum     1230.90
_exptl_crystal_F_000      2272.00
_reflns_d_resolution_high  0.7897

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 'x, -y, z+1/2'
 'x+1/2, y+1/2, z'
 'x+1/2, -y+1/2, z+1/2'
 '-x, -y, -z'
 '-x, y, -z-1/2'
 '-x+1/2, -y+1/2, -z'
 '-x+1/2, y+1/2, -z-1/2'

_cell_length_a    16.9630
_cell_length_b    14.6770
_cell_length_c    18.7760
_cell_angle_alpha  90.000
_cell_angle_beta   95.047
_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
   2   0   0    47557.89    49754.94    243.56 o
   4   0   0    51711.14    43929.61    242.33 o
```

6	0	0	77890.79	72766.55	2314.64	o
8	0	0	2414.97	1828.79	72.44	o
10	0	0	459.86	793.42	113.40	o
12	0	0	10016.98	8273.54	180.07	o
14	0	0	2818.39	2983.65	258.91	o
16	0	0	1894.72	1782.40	288.91	o
18	0	0	139.93	84.54	322.42	o
20	0	0	5486.09	6141.47	439.17	o
1	1	0	39008.44	35726.48	1079.70	o
3	1	0	8319.01	5768.54	212.19	o
5	1	0	3542.65	3217.30	96.32	o
7	1	0	265.71	406.92	45.98	o
9	1	0	13229.66	10886.70	226.33	o
11	1	0	3916.85	4282.09	104.22	o
13	1	0	1383.10	1541.11	113.72	o
15	1	0	3.52	25.20	169.18	o
17	1	0	230.11	147.39	211.09	o
19	1	0	127.15	86.34	258.91	o
21	1	0	1126.31	953.69	264.84	o
0	2	0	21208.02	19124.77	349.95	o
2	2	0	759.21	1040.06	33.16	o
4	2	0	2763.22	3287.99	114.40	o
6	2	0	16829.98	17089.83	550.81	o
8	2	0	3458.27	3647.20	97.39	o
10	2	0	7180.01	6895.76	141.80	o
12	2	0	796.41	762.72	99.53	o
14	2	0	1028.21	994.63	172.96	o
16	2	0	1911.15	2115.17	268.63	o
18	2	0	122.76	37.55	236.87	o
20	2	0	287.95	413.24	226.28	o
1	3	0	11454.13	9872.39	180.23	o
3	3	0	4521.75	2883.12	49.52	o
5	3	0	6653.73	7445.61	209.12	o
7	3	0	1915.26	1465.26	43.99	o
9	3	0	28330.84	29764.75	879.48	o
11	3	0	25.80	60.42	81.97	o
13	3	0	977.21	974.25	154.83	o
15	3	0	125.94	141.79	177.48	o
17	3	0	1624.85	1748.58	221.51	o
19	3	0	56.81	-56.71	193.53	o
0	4	0	127993.21	113369.35	1185.62	o
2	4	0	12959.18	13371.92	289.97	o
4	4	0	3732.82	2406.03	39.84	o
6	4	0	747.33	903.98	46.90	o
8	4	0	3499.00	4203.96	98.14	o
10	4	0	19595.32	21289.95	561.65	o
12	4	0	8364.16	8156.93	152.91	o
14	4	0	4076.42	4423.33	180.53	o
16	4	0	2507.79	2917.32	206.45	o

18	4	0	57.98	28.07	215.00	o
20	4	0	197.32	335.68	280.53	o
1	5	0	4802.92	4320.01	72.62	o
3	5	0	14749.54	12807.98	257.34	o
5	5	0	1776.84	2074.72	47.80	o
7	5	0	1566.07	1529.51	54.65	o
9	5	0	4167.32	4558.16	134.28	o
11	5	0	2208.08	2550.20	91.91	o
13	5	0	547.25	798.87	149.99	o
15	5	0	131.07	-179.05	319.76	o
17	5	0	753.50	699.78	202.06	o
19	5	0	136.90	14.58	252.50	o
0	6	0	23562.41	20321.12	568.22	o
2	6	0	19100.05	21487.22	301.70	o
4	6	0	4596.37	4310.97	58.00	o
6	6	0	124.56	111.91	52.63	o
8	6	0	2883.71	2556.24	84.43	o
10	6	0	3044.80	2686.13	150.39	o
12	6	0	2489.63	2287.09	314.52	o
14	6	0	3078.71	3029.84	317.38	o
16	6	0	720.42	697.46	187.35	o
18	6	0	57.57	-41.55	225.45	o
1	7	0	1876.90	1131.77	51.26	o
3	7	0	7833.32	5586.64	112.89	o
5	7	0	17249.02	15858.47	419.32	o
7	7	0	11541.20	11301.02	193.69	o
9	7	0	5727.11	5596.65	336.28	o
11	7	0	10114.25	10881.74	503.77	o
13	7	0	479.44	458.04	135.00	o
15	7	0	1141.60	955.24	174.74	o
17	7	0	5.57	-75.74	213.92	o
19	7	0	171.95	-13.10	265.18	o
0	8	0	12218.57	9582.26	544.83	o
2	8	0	2408.05	3284.06	88.30	o
4	8	0	168.18	39.31	58.08	o
6	8	0	3.90	-32.76	54.50	o
8	8	0	6108.21	6423.12	240.74	o
10	8	0	29.33	70.07	178.11	o
12	8	0	8339.72	9089.58	165.07	o
14	8	0	5080.04	4878.71	199.02	o
16	8	0	1642.99	1804.81	203.99	o
18	8	0	971.12	1302.32	234.60	o
1	9	0	4488.39	4535.45	95.86	o
3	9	0	96.34	1.37	67.14	o
5	9	0	3048.42	2797.23	128.18	o
7	9	0	147.75	352.05	67.15	o
9	9	0	314.13	113.03	76.93	o
11	9	0	0.74	3.27	101.05	o
13	9	0	533.43	723.34	135.16	o

15	9	0	1414.54	1393.92	172.63	o
17	9	0	2.79	107.17	205.55	o
0	10	0	41510.53	37597.61	1555.63	o
2	10	0	18688.56	18960.86	240.79	o
4	10	0	2166.86	2056.82	92.89	o
6	10	0	233.19	128.23	80.60	o
8	10	0	1.15	23.98	108.98	o
10	10	0	3756.63	4581.53	138.23	o
12	10	0	60.20	102.18	124.73	o
14	10	0	3464.05	4295.52	177.92	o
16	10	0	932.46	974.57	195.19	o
1	11	0	23.78	5.25	85.63	o
3	11	0	6886.90	5936.48	121.58	o
5	11	0	1372.39	1171.22	95.43	o
7	11	0	1979.47	1776.35	108.28	o
9	11	0	9427.30	9400.50	475.75	o
11	11	0	137.48	178.89	141.98	o
13	11	0	41.34	44.51	167.86	o
15	11	0	74.31	241.60	189.87	o
0	12	0	523.34	645.53	133.88	o
2	12	0	2031.60	1896.07	108.15	o
4	12	0	3398.91	3102.58	114.48	o
6	12	0	1957.74	2192.58	123.65	o
8	12	0	560.47	827.70	146.81	o
10	12	0	1237.12	988.70	161.78	o
12	12	0	470.68	591.89	159.79	o
14	12	0	2.49	-34.22	206.00	o
16	12	0	37.61	-23.79	229.18	o
1	13	0	2679.01	2969.19	125.41	o
3	13	0	1222.75	961.62	110.07	o
5	13	0	2506.31	2445.04	125.73	o
7	13	0	1018.04	1150.48	156.40	o
9	13	0	1573.54	890.82	163.47	o
11	13	0	978.40	1555.95	180.43	o
13	13	0	0.38	-42.59	216.30	o
0	14	0	21102.94	17986.64	281.65	o
2	14	0	3180.85	3790.16	138.45	o
4	14	0	2202.78	1956.56	126.89	o
6	14	0	582.18	476.46	136.17	o
8	14	0	297.02	102.99	179.76	o
10	14	0	1333.32	1672.81	184.38	o
12	14	0	1490.09	1337.77	192.35	o
1	15	0	2771.86	2632.90	140.77	o
3	15	0	1435.68	1231.02	137.83	o
5	15	0	432.16	463.31	143.27	o
7	15	0	1009.68	1039.80	202.04	o
9	15	0	2047.40	1854.92	213.04	o
11	15	0	468.58	698.44	208.99	o
0	16	0	218.36	217.82	197.72	o

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  2  16   0     237.52     252.87    141.37 o
  4  16   0     887.60     939.76    164.66 o
  6  16   0     357.84     561.60    223.96 o
  8  16   0     402.39     430.82    213.70 o
 10  16   0     553.80     979.95    461.55 o
  1  17   0      62.92     264.68    160.95 o
  3  17   0     124.33      -7.79    164.12 o
  5  17   0     835.38     970.62    189.54 o
  7  17   0     320.81     627.23    254.04 o
  0  18   0     487.29     259.80    254.17 o
  2  18   0     891.43    1204.30    184.27 o
  4  18   0     265.88     405.62    335.14 o
-21   1   1     230.44     472.28    302.79 o
-19   1   1    1941.48    1972.29    258.26 o
-17   1   1    1248.05    1581.99    217.97 o
-15   1   1     859.99     823.60    127.10 o
-13   1   1      44.14      -2.34    100.29 o
-11   1   1      33.02     -36.42     85.83 o
 -9   1   1     343.11     213.82     58.66 o
 -7   1   1     180.16     190.43     43.43 o
 -5   1   1   29224.18   31110.11    404.70 o
 -3   1   1   50111.97   44561.93    526.33 o
 -1   1   1   38733.86   42356.93   1215.00 o
  1   1   1      68.27     138.54     17.35 o
  3   1   1   15639.84   15092.95    503.78 o
  5   1   1    1040.91     724.88     34.94 o
  7   1   1    1253.94    1232.76     50.49 o
  9   1   1    6192.52    5454.42     90.97 o
 11   1   1   16472.28   16329.65    314.00 o
 13   1   1   10136.83    9981.90    207.85 o
 15   1   1    2792.91    2946.56    199.74 o
 17   1   1      30.01       3.68    209.62 o
 19   1   1     337.09     283.56    181.25 o
-20   2   1    1152.39    1456.94    278.90 o
-18   2   1       7.00      26.03    234.86 o
-16   2   1     496.78     295.75    190.79 o
-14   2   1     766.90     746.90    117.42 o
-12   2   1   23000.62   21482.89    417.35 o
-10   2   1    6648.26    7204.44    113.16 o
 -8   2   1    4856.78    4049.80     71.63 o
 -6   2   1     527.42     639.13     47.14 o
 -4   2   1    5541.88    4926.40    109.93 o
 -2   2   1   28755.35   34523.60    360.45 o
  0   2   1   43132.04   35111.85   1295.80 o
  2   2   1   11725.85   16251.77    329.23 o
  4   2   1   98717.72   92031.55   1261.09 o
  6   2   1   12567.91   12868.83    233.17 o
  8   2   1     473.86     630.25     62.71 o
 10   2   1    8936.98    9806.65    145.31 o
```

12	2	1	1702.83	1753.94	219.44	o
14	2	1	2632.32	2036.13	168.84	o
16	2	1	5030.22	5506.03	392.23	o
18	2	1	41.67	-145.29	186.16	o
20	2	1	1017.04	918.73	201.12	o
-19	3	1	230.72	293.67	422.36	o
-17	3	1	507.72	727.31	225.78	o
-15	3	1	634.77	761.02	128.48	o
-13	3	1	254.38	174.12	101.91	o
-11	3	1	727.66	626.59	87.58	o
-9	3	1	10240.22	9202.12	145.19	o
-7	3	1	38.55	58.99	49.59	o
-5	3	1	39.59	85.22	58.07	o
-3	3	1	4427.24	2426.93	59.31	o
-1	3	1	24023.11	20042.59	318.62	o
1	3	1	202.41	312.43	26.81	o
3	3	1	14694.68	12281.64	176.80	o
5	3	1	2021.71	2460.77	78.86	o
7	3	1	4319.61	5432.09	89.60	o
9	3	1	1455.94	1203.14	76.51	o
11	3	1	7880.07	7948.91	171.57	o
13	3	1	1149.79	1088.40	152.55	o
15	3	1	177.05	267.84	173.18	o
17	3	1	118.07	152.53	167.49	o
19	3	1	558.75	654.10	204.82	o
-20	4	1	0.09	-91.60	484.88	o
-18	4	1	524.13	695.26	418.55	o
-16	4	1	1684.08	1916.40	198.34	o
-14	4	1	988.83	935.61	113.43	o
-12	4	1	8242.52	7779.69	366.01	o
-10	4	1	37883.63	38114.65	879.34	o
-8	4	1	205.28	170.79	50.03	o
-6	4	1	1159.22	1230.38	81.94	o
-4	4	1	5389.05	3865.65	107.24	o
-2	4	1	5613.44	5202.41	86.72	o
0	4	1	190.81	463.70	35.97	o
2	4	1	121.98	51.78	33.04	o
4	4	1	3091.78	3250.91	74.69	o
6	4	1	683.55	575.83	41.65	o
8	4	1	4589.94	5137.08	149.68	o
10	4	1	16766.80	18473.16	466.70	o
12	4	1	1448.98	1474.51	142.34	o
14	4	1	2959.26	3098.91	179.96	o
16	4	1	490.49	564.02	151.63	o
18	4	1	362.06	247.19	229.53	o
20	4	1	36.69	13.45	246.29	o
-19	5	1	235.05	254.16	435.50	o
-17	5	1	645.81	846.36	381.87	o
-15	5	1	391.74	465.71	284.08	o

-13	5	1	389.35	268.08	93.09	o
-11	5	1	348.19	369.14	60.58	o
-9	5	1	15010.11	14009.33	244.56	o
-7	5	1	24538.87	24172.07	179.59	o
-5	5	1	21046.06	18467.62	255.76	o
-3	5	1	13259.67	12121.12	175.47	o
-1	5	1	73452.40	70470.59	898.87	o
1	5	1	44653.24	41008.25	836.87	o
3	5	1	2762.10	2943.79	79.68	o
5	5	1	10977.86	8865.66	106.26	o
7	5	1	3847.73	4188.76	62.69	o
9	5	1	4255.63	4342.76	94.25	o
11	5	1	527.49	476.34	111.35	o
13	5	1	1815.23	2119.22	119.96	o
15	5	1	12.16	-48.33	134.16	o
17	5	1	3.33	-47.27	212.58	o
19	5	1	868.48	704.22	258.26	o
-18	6	1	133.89	264.56	218.59	o
-16	6	1	936.42	710.96	381.90	o
-14	6	1	1815.33	2285.76	310.43	o
-12	6	1	7939.67	6906.32	477.89	o
-10	6	1	11986.66	12497.36	247.77	o
-8	6	1	11010.21	10663.59	152.43	o
-6	6	1	8228.04	7158.69	124.05	o
-4	6	1	4511.40	5071.60	101.46	o
-2	6	1	4569.65	3869.44	136.75	o
0	6	1	27018.72	21892.22	365.17	o
2	6	1	4094.93	5228.95	141.98	o
4	6	1	28141.62	24749.43	395.20	o
6	6	1	3028.24	3192.99	59.94	o
8	6	1	177.89	156.40	49.89	o
10	6	1	344.21	350.49	65.83	o
12	6	1	5531.21	5389.88	124.28	o
14	6	1	2928.34	2176.40	173.65	o
16	6	1	2629.99	2961.10	217.82	o
18	6	1	730.67	626.90	248.72	o
-19	7	1	6.74	-56.84	256.17	o
-17	7	1	2.24	62.59	202.93	o
-15	7	1	23.68	18.60	379.81	o
-13	7	1	2.25	-23.58	291.47	o
-11	7	1	297.98	197.26	195.92	o
-9	7	1	1746.93	1484.68	172.22	o
-7	7	1	516.08	252.14	86.89	o
-5	7	1	2409.84	3085.94	68.37	o
-3	7	1	8264.90	7350.19	144.99	o
-1	7	1	28136.02	31324.41	498.53	o
1	7	1	247.79	726.30	53.86	o
3	7	1	33.73	61.08	55.38	o
5	7	1	8725.05	8680.09	157.76	o

```
  7   7   1     262.77     172.82     51.06 o
  9   7   1     712.22     661.21     59.90 o
 11   7   1     856.98    1071.35    109.17 o
 13   7   1      18.18     -73.86    143.09 o
 15   7   1     669.09     873.37    185.01 o
 17   7   1     163.94      79.69    217.35 o
 19   7   1     227.23     256.45    265.41 o
-18   8   1      65.97     -24.95    222.03 o
-16   8   1     113.98      -3.08    189.78 o
-14   8   1     246.74     157.41    148.94 o
-12   8   1    2940.12    3263.33    388.89 o
-10   8   1    9515.45   10480.60    591.67 o
 -8   8   1       9.78      25.73    152.95 o
 -6   8   1    1243.28    1602.96     61.33 o
 -4   8   1       7.88      -9.49     52.73 o
 -2   8   1    7224.51    6010.63     94.09 o
  0   8   1      82.21      43.12     59.00 o
  2   8   1   13940.58   13203.01    264.44 o
  4   8   1   17871.63   16230.83    329.87 o
  6   8   1   10601.23   10877.14    182.88 o
  8   8   1     498.84     486.96     66.19 o
 10   8   1     469.41     576.76    100.13 o
 12   8   1     335.05     224.32    129.31 o
 14   8   1    1083.38    1443.96    165.11 o
 16   8   1     264.43     239.74    186.67 o
 18   8   1     373.77     289.89    229.33 o
-17   9   1     814.15    1154.56    202.35 o
-15   9   1     169.92     176.80    162.98 o
-13   9   1    1194.96    1163.16    365.47 o
-11   9   1      85.43      98.43    195.38 o
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 5   1  22     118.82     145.51    494.22 o
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-6   2  22      36.30     139.36    324.41 o
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-5   3  22     321.17     227.87    351.05 o
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-1   3  22     351.50     494.20    374.41 o
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 3   3  22    2030.17    2151.48    368.43 o
 5   3  22    1709.13    1858.16    415.33 o
-8   4  22     313.46     495.07    574.01 o
-6   4  22     589.87     666.71    358.39 o
-4   4  22    1972.93    2161.35    352.66 o
-2   4  22    3621.83    2837.36    387.41 o
 0   4  22    1208.30     987.95    498.50 o
 2   4  22     315.17     112.63    341.02 o
 4   4  22      13.10    -130.54    358.87 o
-7   5  22      14.23      19.55    584.21 o
-5   5  22      76.23     -18.00    361.93 o
-3   5  22     140.10     -75.80    349.19 o
-1   5  22      11.92     176.86    482.58 o
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-6   6  22     211.88      -3.32    542.14 o
-2   6  22     906.05     930.03    488.88 o
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 0   2  23    1133.43    1107.37    388.47 o
 2   2  23    1378.02    1050.13    595.34 o
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  -5   3  23      245.14       80.23    596.92 o
  -3   3  23     1171.13     1020.80    363.64 o
  -1   3  23      168.83     -152.79    401.10 o
   1   3  23        7.48     -274.77    550.99 o
  -4   4  23       41.84      310.86    543.39 o
  -2   4  23      180.79      164.78    406.81 o
   0   4  23        1.47      -85.67    545.20 o

===End of fcf
#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_{[Ni(tpt)Cl(H2O)2]Cl + [Ni(tpt)Cl2(H2O)]}.4H2O, 2.8 + 2.9 (B)
_shelx_title ' 2.8 + 2.9 (B) in P-1'
_shelx_refln_list_code          4
_shelx_F_calc_maximum      573.84
_exptl_crystal_F_000      1036.00
_reflns_d_resolution_high  0.7992

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, -y, -z'

_cell_length_a    13.0080
_cell_length_b    13.3765
_cell_length_c    13.9985
_cell_angle_alpha  85.304
_cell_angle_beta   67.460
_cell_angle_gamma  69.039

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
   1   0   0     7674.73     7448.49    184.66 o
   2   0   0    14791.81    14877.46    259.94 o
   3   0   0     1283.19     1321.42     26.40 o
   4   0   0     1282.24     1198.16     26.23 o
   5   0   0    13073.17    12947.56    228.04 o
   6   0   0       71.86       59.08     14.02 o
   7   0   0     6319.35     5986.55    108.93 o
   8   0   0     1569.30     1565.02     43.49 o
```

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 -8   1   0     381.85     397.91     24.76 o
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 -5   1   0     785.90     901.21     32.57 o
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  4   1   0   13480.46   13218.12    240.23 o
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 -3   2   0   18170.30   17632.65    308.74 o
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 -1   2   0    1593.33    1476.99     27.65 o
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 6  12   0     528.03     604.44    36.12 o
 7  12   0    3066.90    3129.15    96.36 o
 8  12   0       0.07      13.65    39.37 o
 9  12   0     185.67     118.40    67.47 o
10  12   0     567.47     585.62    79.25 o
11  12   0    3545.42    3459.64   110.18 o
12  12   0      37.31      88.84    53.62 o
13  12   0     853.61     777.78    64.57 o
-4  13   0     822.77     929.16    65.88 o
-3  13   0     849.17     933.03   115.22 o
-2  13   0     132.99      87.65    46.45 o
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 6  13   0     151.58     133.64    35.16 o
 7  13   0    1122.80    1046.61    59.33 o
 8  13   0    1372.30    1181.52    60.30 o
 9  13   0    3556.19    3257.53   138.77 o
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11  13   0      47.08       5.72    54.32 o
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 3  15   0     460.64     413.29    57.29 o
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 -8 -15   1    1300.29    1258.26     97.39 o
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 -4 -15   1       3.41       1.38     55.49 o
 -3 -15   1      36.31      70.13     63.34 o
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 5  14   2     914.44     998.91     99.30 o
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 6  15   2     254.69     228.68     59.04 o
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 9  15   2      63.40       4.22     74.32 o
10  15   2     652.01     761.52     64.92 o
11  15   2     200.47     144.50     78.93 o
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 -7 -11   5      331.37      390.60     51.93 o
 -6 -11   5      786.45      949.41     67.79 o
 -5 -11   5        0.39      -13.37     54.42 o
 -4 -11   5       36.27      -21.73     61.90 o
 -3 -11   5     1127.88     1182.26     52.52 o
 -2 -11   5      289.61      284.61     53.31 o
 -1 -11   5     7126.59     6900.49    245.07 o
  0 -11   5      184.90      156.46     44.07 o
  1 -11   5      599.41      604.14     38.95 o
  2 -11   5       89.79       70.51     32.96 o
  3 -11   5      131.27      172.92     37.53 o
  4 -11   5     1179.11     1155.37     56.20 o
  5 -11   5        7.04       24.49     36.80 o
  6 -11   5     1167.05     1170.93     82.75 o
  7 -11   5      151.38      106.62     69.38 o
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 -9 -10   5      919.44      991.32     62.21 o
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 -5 -10   5     4191.18     4253.41    160.09 o
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  2 -10   5      701.95      698.56     35.14 o
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 -7  -7   6     247.69     253.07     34.47 o
 -6  -7   6     180.75     188.24     31.56 o
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-5   9   6     184.15     215.85     53.33 o
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 6   9  10    1198.40    1201.09     56.81 o
 7   9  10    2485.86    2449.29    121.26 o
 8   9  10     106.09     117.53     33.53 o
 9   9  10      27.42      36.30     31.71 o
10   9  10     258.67     259.60     35.48 o
11   9  10     206.54     252.58     45.83 o
12   9  10     541.50     518.30     42.62 o
13   9  10    1185.40    1233.14     56.23 o
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-1  10  10      32.37     -39.87     83.71 o
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 3  10  10     696.69     846.97     58.80 o
 4  10  10     124.76     126.70     41.64 o
 5  10  10     477.26     498.32     75.59 o
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 7  10  10     159.62     136.21     38.13 o
 8  10  10     384.10     368.78     37.53 o
 9  10  10      70.84      78.52     37.11 o
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14  10  10     316.62     276.95     48.49 o
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 9  12  10    470.89    542.04    48.67 o
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12  12  10    141.19     66.06    51.51 o
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 4  13  10    390.48    321.14    76.07 o
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 5  -7  11     295.84     312.25     36.08 o
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 5   6  11    4373.84    4084.91    142.27 o
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14	5	12	104.33	120.63	52.20	o
15	5	12	103.32	88.48	62.06	o
-3	6	12	146.82	137.09	77.34	o
-2	6	12	187.57	277.05	55.01	o
-1	6	12	199.07	153.31	50.87	o
0	6	12	258.76	262.57	43.77	o
1	6	12	747.46	824.88	67.79	o
2	6	12	2569.51	2530.10	80.77	o
3	6	12	665.41	754.23	43.13	o
4	6	12	28.07	-0.54	32.65	o
5	6	12	20.90	-8.08	49.81	o
6	6	12	8059.51	7347.08	189.28	o
7	6	12	5465.90	5287.71	138.53	o

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 8   6  12      27.07     111.40     38.83 o
 9   6  12     330.30     330.37     41.69 o
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11   6  12     371.16     443.99     46.15 o
12   6  12      14.18     113.31     46.15 o
13   6  12       3.96      -9.55     47.42 o
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-2   7  12     134.57     128.16     56.58 o
-1   7  12     228.70     271.19     56.10 o
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 3   7  12      81.65     135.60     89.43 o
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 6   7  12     156.66     209.86     34.21 o
 7   7  12    2631.59    2615.15    185.87 o
 8   7  12      80.95     127.86     31.20 o
 9   7  12      10.47     -35.96     41.69 o
10   7  12       5.45     -21.96     44.24 o
11   7  12      32.83       8.91     44.56 o
12   7  12     750.36     774.36     60.47 o
13   7  12       0.63     -49.97     49.97 o
14   7  12     106.19     121.58     60.47 o
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 4   8  12     129.86     125.04     37.62 o
 5   8  12     140.20     114.68     36.85 o
 6   8  12       8.14     -29.08     35.52 o
 7   8  12     131.62      41.60     60.63 o
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10   8  12      35.89      22.05     34.80 o
11   8  12      68.47     113.94     51.88 o
12   8  12     182.98     119.35     56.65 o
13   8  12     277.01     373.02     64.93 o
14   8  12      63.41      25.14     62.06 o
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 7   9  12      69.84      62.59     37.46 o
 8   9  12       3.78       6.13     41.38 o
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 7  13  12      210.35      238.39     95.16 o
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 5  -8  13     260.78     239.80     43.90 o
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10   9  13      85.92      31.19     62.38 o
11   9  13      14.55       5.41     57.61 o
12   9  13     185.34     119.67     63.97 o
13   9  13     605.25     565.57     73.20 o
 3  10  13      79.73       4.44     54.40 o
 4  10  13      49.68      36.99     50.93 o
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11  10  13      97.73     122.85     70.98 o
12  10  13      83.81     203.38     73.20 o
 4  11  13     540.35     735.21    105.35 o
 5  11  13     127.59      -6.34     57.49 o
 6  11  13     138.39      42.64     57.61 o
 7  11  13      18.33      38.97     52.18 o
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 8  11  13       4.49       10.61    108.21 o
 9  11  13     244.57      299.92     49.49 o
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11  11  13     994.29     1042.36     75.27 o
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 8  12  13      82.73      124.45     56.90 o
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 2  -8  14     772.78      830.01     56.55 o
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 4  -8  14    1027.22     1004.66     63.97 o
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 5  -7  14      19.58       59.84     61.11 o
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 5   4  15     153.38     197.09     67.47 o
 6   4  15       0.13     -35.01     50.92 o
 7   4  15      19.52      11.14     47.10 o
 8   4  15      64.25     -42.01     42.01 o
 9   4  15       2.94      -5.41     42.33 o
10   4  15      48.86      93.57     40.10 o
11   4  15     199.36     198.28     41.38 o
12   4  15     124.16      91.66     40.10 o
13   4  15     272.45     241.57     45.51 o
 1   5  15      61.59      68.88     55.90 o
 2   5  15     384.90     425.54     51.23 o
 3   5  15     107.54     120.38     46.16 o
 4   5  15      45.21      36.29     42.45 o
 5   5  15      86.09      63.14     38.52 o
 6   5  15       6.57     -25.46     55.06 o
 7   5  15      99.89      35.96     54.42 o
 8   5  15     176.41     146.09     52.83 o
 9   5  15       2.55      66.20     46.79 o
10   5  15      14.31       5.73     44.24 o
11   5  15       1.83      34.37     45.51 o
12   5  15    1280.11    1246.04     70.02 o
13   5  15     259.87     239.66     49.65 o
 1   6  15      69.19     148.00     96.12 o
 2   6  15     330.88     261.45     56.04 o
 3   6  15      16.90      63.31     88.96 o
 4   6  15      10.77      -7.02     43.82 o
```

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 5   6  15       8.74     -33.50     40.91 o
 6   6  15     103.68      104.52     52.99 o
 7   6  15    1830.75     1601.87     89.75 o
 8   6  15      38.81      164.23     57.61 o
 9   6  15     267.71      369.83     55.06 o
10   6  15      32.61      -49.33     49.33 o
11   6  15    1385.90     1521.35     82.11 o
12   6  15     147.07      128.58     51.56 o
 2   7  15      18.24       -6.47     59.33 o
 3   7  15     120.38      191.46    121.58 o
 4   7  15      27.46       27.90     47.62 o
 5   7  15      53.50       27.75     46.79 o
 6   7  15      12.97       24.84     45.63 o
 7   7  15     615.93      621.91     70.98 o
 8   7  15    3145.01     2991.77    137.18 o
 9   7  15      46.12      115.53     56.02 o
10   7  15     231.24      352.65     58.88 o
11   7  15      16.00       -4.14     58.24 o
12   7  15       8.79      115.22     57.93 o
 4   8  15      72.61       36.12     81.96 o
 5   8  15      98.79       44.70     72.88 o
 6   8  15     242.96      260.21     51.97 o
 7   8  15    1119.07      989.99     58.59 o
 8   8  15       0.07      -55.38     62.38 o
 9   8  15     981.09      775.63     74.79 o
10   8  15     272.76      116.81     61.11 o
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 5   9  15     563.00      574.16     74.00 o
 6   9  15     165.95      141.74     49.75 o
 7   9  15     406.41      349.09     53.92 o
 8   9  15     640.46      430.94     77.02 o
 9   9  15      28.02       63.97     66.84 o
 2  -5  16     445.03      485.05     73.52 o
 1  -4  16       0.89        7.60     47.59 o
 2  -4  16     889.37      998.68     59.83 o
 3  -4  16     590.18      601.86     78.61 o
 4  -4  16     269.63      412.48     75.75 o
 5  -4  16     740.83      646.73     69.07 o
 6  -4  16       4.68       -2.55     68.43 o
 7  -4  16     356.98      390.20     60.79 o
 0  -3  16     989.36      903.23     67.27 o
 1  -3  16      72.36      147.21     50.47 o
 2  -3  16       1.83       10.05    112.99 o
 3  -3  16       1.88      -67.79     67.79 o
 4  -3  16     365.65      335.14     70.98 o
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 6  -3  16     603.91      598.04     59.84 o
 7  -3  16       6.75      -12.09     51.24 o
 8  -3  16     480.74      445.58     62.06 o
```

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 3  -2  16     922.76    1215.81     92.62 o
 4  -2  16      60.15      63.02     64.61 o
 5  -2  16     204.90     223.43     61.11 o
 6  -2  16     194.45     259.71     59.20 o
 7  -2  16     270.87     242.21     49.01 o
 8  -2  16       4.57      37.56     45.83 o
 9  -2  16     174.12     138.45     52.83 o
 0  -1  16      26.48      24.72     56.21 o
 1  -1  16     640.06     740.23     58.39 o
 2  -1  16     707.43     760.10     53.04 o
 3  -1  16      11.96     -37.34     44.07 o
 4  -1  16       0.18     -59.52     59.52 o
 5  -1  16      47.33      29.28     56.97 o
 6  -1  16      63.28     114.26     53.47 o
 7  -1  16      22.61      -0.95     49.01 o
 8  -1  16     451.22     415.67     50.61 o
 9  -1  16      24.62      48.70     43.92 o
10  -1  16       7.81     -32.78     45.51 o
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 2   0  16      11.51      15.53     46.36 o
 3   0  16     120.32     130.44     45.39 o
 4   0  16     268.43     220.25     66.52 o
 5   0  16     289.56     355.83     65.88 o
 6   0  16      36.49      58.88     53.15 o
 7   0  16     396.94     336.73     51.88 o
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 4   1  16      51.61       1.59     57.93 o
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 3   2  16     137.45     157.07     45.22 o
 4   2  16      91.40      10.18     58.56 o
 5   2  16      46.08     -12.41     54.42 o
 6   2  16     227.83     281.04     52.52 o
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 8   2  16      91.62     146.41     45.19 o
```

```
 9   2  16     271.74     258.76     42.33 o
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 3   3  16     102.62      80.30     70.98 o
 4   3  16     157.65     192.98     61.90 o
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 2   4  16      20.61     -54.43     54.43 o
 3   4  16      17.77     -35.30     83.55 o
 4   4  16     262.41     197.30     47.65 o
 5   4  16       1.23      51.88     63.65 o
 6   4  16     387.26     525.15     66.84 o
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 8   4  16     120.70     146.41     50.29 o
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11   4  16     499.59     491.41     47.42 o
 3   5  16     274.41     273.43     62.22 o
 4   5  16      78.15      80.05     71.77 o
 5   5  16     473.19     548.07     74.16 o
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 7   5  16       1.69      12.73     58.88 o
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 9   5  16      53.72     123.49     53.15 o
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11   5  16       5.58      64.93     49.33 o
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 7   6  16     297.73     254.62     70.98 o
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 5   7  16     142.06     180.28     54.68 o
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 7   7  16     203.23     249.21     71.61 o
 8   7  16      29.97       8.28     68.43 o
 9   7  16     542.28     426.17     65.88 o
 4  -1  17       6.12      77.02     72.57 o
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 6  -1  17      83.06     154.68     64.29 o
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 4   0  17     239.07     194.15     70.66 o
 5   0  17      71.35      46.79     69.70 o
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   7   0  17      11.53      60.79     58.88 o
   8   0  17       3.47     -53.15     53.15 o
   3   1  17     132.05     110.44     65.25 o
   4   1  17      45.94      95.16     67.16 o
   5   1  17     252.17     202.74     64.93 o
   6   1  17      26.81      -5.09     59.52 o
   7   1  17      72.81      80.84     55.70 o
   8   1  17      67.17      56.65     51.56 o
   9   1  17     444.92     486.96     54.42 o
   3   2  17      47.00      92.94     68.75 o
   4   2  17     421.81     397.84     71.29 o
   5   2  17       8.68      60.79     59.20 o
   6   2  17     837.79     787.73     71.29 o
   7   2  17       0.63      81.16     55.38 o
   8   2  17     461.17     494.92     55.70 o
   9   2  17       6.06     101.21     47.74 o
   5   3  17     291.26     299.18     73.20 o
   6   3  17     253.11     212.93     64.61 o
   7   3  17     109.04     113.62     59.52 o
   8   3  17     122.44     176.96     55.06 o
   9   3  17      23.35      -0.32     50.61 o
   7   4  17       1.46      63.65     64.93 o
   8   4  17      79.39     -36.60     57.61 o
   9   4  17      13.67      50.61     56.97 o

===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_[Ni(Htpt)Cl(H2O)2]Cl2.2H2O, 2.10 (C)
_shelx_title ' 2.10 (C) in P2(1)/n'
_shelx_refln_list_code          4
_shelx_F_calc_maximum      671.12
_exptl_crystal_F_000      1128.00
_reflns_d_resolution_high  0.7980

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x+1/2, y+1/2, -z+1/2'
 '-x, -y, -z'
 'x-1/2, -y-1/2, z-1/2'

_cell_length_a    12.4599
_cell_length_b    14.5521
_cell_length_c    13.3774
_cell_angle_alpha  90.000
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_cell_angle_beta  107.368
_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
  2   0   0     922.33     1119.65     14.13 o
  4   0   0   42922.13    42537.42    739.57 o
  6   0   0   23873.29    25070.39    394.28 o
  8   0   0     772.61      809.63     16.22 o
 10   0   0    1245.76     1295.76     43.37 o
 12   0   0       8.52       10.16     12.46 o
 14   0   0      77.64      107.47     16.52 o
  1   1   0    1915.45     1996.88     36.80 o
  2   1   0    3145.09     3111.31     30.11 o
  3   1   0    4402.51     4432.44     43.23 o
  4   1   0    7897.35     7779.56     63.40 o
  5   1   0    4306.49     4767.40     41.18 o
  6   1   0     210.56      156.75      4.34 o
  7   1   0    1262.23     1349.86     14.55 o
  8   1   0     769.30      823.52     12.31 o
  9   1   0     814.93      822.48     18.61 o
 10   1   0     380.14      371.18     14.29 o
 11   1   0       4.02       13.00      7.30 o
 12   1   0    1195.10     1075.42     23.12 o
 13   1   0      20.84       35.32     11.61 o
 14   1   0      49.84       75.06     11.15 o
  0   2   0    6233.91     6308.13    131.40 o
  1   2   0   16552.73    16084.80    137.89 o
  2   2   0      85.99       91.99      2.79 o
  3   2   0   16083.38    16437.88    130.35 o
  4   2   0     186.85      198.95      3.62 o
  5   2   0   11018.83    11677.75    100.93 o
  6   2   0    1630.81     1598.80     15.35 o
  7   2   0       6.75       13.63      3.42 o
  8   2   0    1124.73     1137.78     17.77 o
  9   2   0    2461.76     2426.21     30.44 o
 10   2   0     771.99      753.94     17.18 o
 11   2   0     794.16      909.39     20.06 o
 12   2   0     568.98      547.45     18.52 o
 13   2   0    2531.08     2543.85     42.76 o
 14   2   0       3.60       11.07     14.73 o
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 2   3   0      84.52     112.73      2.72 o
 3   3   0    2545.28    2724.47     23.24 o
 4   3   0     202.29     206.21      4.50 o
 5   3   0    3151.95    3155.58     26.99 o
 6   3   0     406.10     500.67      7.26 o
 7   3   0    3838.26    3609.71     35.49 o
 8   3   0      16.25      23.56      5.21 o
 9   3   0    2481.77    2452.96     30.92 o
10   3   0     836.20     791.38     14.69 o
11   3   0    2210.02    2082.13     34.88 o
12   3   0      25.08      51.71      8.63 o
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14   3   0      10.12      35.33     21.14 o
 0   4   0   10955.59   11185.73    124.18 o
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 2   4   0    7611.04    7603.60     55.25 o
 3   4   0   23129.58   23206.90    169.27 o
 4   4   0    2658.58    2900.84     25.74 o
 5   4   0     136.84     176.44      3.85 o
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 1   5   0    5305.18    5465.86     42.15 o
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3	7	0	27.67	7.13	2.97 o
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5	7	0	487.07	598.28	9.43 o
6	7	0	1428.82	1420.54	17.14 o
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8	7	0	413.55	434.66	10.31 o
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 -6   4  15      11.39      51.57     11.36 o
 -5   4  15    1308.09    1261.24     35.15 o
 -4   4  15    1028.45    1072.83     23.98 o
 -3   4  15    2145.77    2046.13     71.36 o
 -2   4  15     804.32     843.66     23.62 o
 -1   4  15     511.87     557.32     21.13 o
  0   4  15    1762.73    1803.08     35.33 o
  1   4  15     108.81     123.90     16.85 o
 -9   5  15     615.14     633.52     25.69 o
 -8   5  15      15.26      19.98     12.47 o
 -7   5  15      77.09     103.90     13.50 o
 -6   5  15       5.15       8.08     11.82 o
 -5   5  15       0.02      24.86     11.24 o
 -4   5  15      50.05      66.62     13.88 o
 -3   5  15     284.84     294.99     16.10 o
 -2   5  15      11.50      44.02     12.48 o
 -1   5  15     570.40     609.38     20.94 o
  0   5  15     672.50     662.76     21.26 o
  1   5  15      20.55     -19.38     19.38 o
 -8   6  15      55.39      65.26     15.65 o
 -7   6  15     250.76     239.63     15.69 o
 -6   6  15     211.48     230.92     15.97 o
 -5   6  15     751.64     768.54     25.96 o
 -4   6  15      13.23      31.36     14.11 o
```

```
-3   6  15      560.11      582.91     20.21 o
-2   6  15      437.68      448.96     18.40 o
-1   6  15       13.38       26.69     12.11 o
 0   6  15       87.33       99.09     16.02 o
-7   7  15       88.32      102.57     18.40 o
-6   7  15       78.54       94.21     19.64 o
-5   7  15      211.04      262.63     20.33 o
-4   7  15       17.73       33.27     13.09 o
-3   7  15       42.68       62.89     12.40 o
-2   7  15        6.45       21.38     11.20 o
-1   7  15       63.58       82.13     12.94 o
-8   0  16      916.65      704.40     32.76 o
-6   0  16       37.06        8.30     14.53 o
-4   0  16       55.77       56.84     16.41 o
-2   0  16       15.54       25.42     27.81 o
-8   1  16     2436.96     2171.51     46.41 o
-7   1  16      557.91      516.88     19.51 o
-6   1  16      182.40      174.89     13.21 o
-5   1  16       10.08       32.08     12.76 o
-4   1  16       23.09       19.98     13.82 o
-3   1  16        2.34       16.68     11.31 o
-2   1  16      170.48      193.08     16.47 o
-8   2  16      132.70      150.84     17.71 o
-7   2  16      322.01      301.37     15.66 o
-6   2  16      394.31      397.97     26.25 o
-5   2  16       28.69       17.73     11.84 o
-4   2  16      667.46      670.04     20.23 o
-3   2  16      100.68      115.13     17.91 o
-2   2  16      534.55      518.14     36.34 o
-1   2  16      149.02      179.10     25.52 o
-8   3  16       21.49      -35.89     35.89 o
-7   3  16       61.57       69.89     15.73 o
-6   3  16        5.09       12.44     11.08 o
-5   3  16      771.14      764.33     21.43 o
-4   3  16        6.21       12.76     13.82 o
-3   3  16      478.35      457.31     21.46 o
-2   3  16       46.10       53.04     15.50 o
-1   3  16     1031.11     1027.42     57.57 o
-7   4  16       44.18       61.68     23.92 o
-6   4  16     1121.47     1036.00     27.29 o
-5   4  16      345.37      365.18     16.87 o
-4   4  16     1224.19     1230.04     34.56 o
-3   4  16      147.99      145.71     40.41 o
-2   4  16     1055.31     1034.24     43.46 o
-6   5  16      573.78      572.32     36.68 o
-5   5  16       88.35      119.25     22.89 o
-4   5  16        1.50       42.61     24.97 o
-3   5  16       66.57       79.21     23.03 o
```

```
===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_[Ni(tpt)(H2O)3](NO3)2, 2.12 (D)
_shelx_title ' 2.12 (D) in P-1'
_shelx_refln_list_code          4
_shelx_F_calc_maximum      317.00
_exptl_crystal_F_000       564.00
_reflns_d_resolution_high  0.7963

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, -y, -z'

_cell_length_a     7.4053
_cell_length_b    13.3116
_cell_length_c    13.8265
_cell_angle_alpha  63.474
_cell_angle_beta   77.848
_cell_angle_gamma  84.599

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
   1   0   0    14424.26    12872.32     55.63 o
   2   0   0     1294.71     1183.21     17.20 o
   3   0   0      630.77      686.13      7.88 o
   4   0   0     2265.61     2186.75     16.93 o
   5   0   0     1031.11     1099.21      8.81 o
   6   0   0      720.40      611.39     13.98 o
   7   0   0      102.79      110.49     12.34 o
   8   0   0        4.69        2.47     12.06 o
   9   0   0       34.22       23.85     15.90 o
  -8   1   0       90.30       77.86     14.53 o
  -7   1   0      254.63      265.12     14.80 o
  -6   1   0      195.12      149.07      7.36 o
  -5   1   0      191.23      238.83      5.71 o
  -4   1   0        0.53        1.43      2.81 o
  -3   1   0      221.26      247.46      4.34 o
```

-2	1	0	1227.90	1024.59	4.54 o
-1	1	0	1894.60	2026.95	13.78 o
0	1	0	6306.60	5873.91	168.34 o
1	1	0	341.58	456.50	4.48 o
2	1	0	1205.00	1298.12	11.00 o
3	1	0	4286.79	4327.16	36.05 o
4	1	0	3219.87	2929.94	21.40 o
6	1	0	4.76	13.43	7.68 o
7	1	0	56.43	55.38	10.14 o
8	1	0	109.46	115.97	14.26 o
9	1	0	162.37	147.78	17.82 o
-8	2	0	2.86	0.00	15.35 o
-7	2	0	59.22	55.38	12.89 o
-6	2	0	0.77	0.72	7.30 o
-5	2	0	1029.88	979.94	20.97 o
-4	2	0	3619.09	3597.04	15.72 o
-3	2	0	797.04	787.24	12.71 o
-2	2	0	3647.94	3661.98	24.26 o
-1	2	0	2680.26	2828.84	24.20 o
0	2	0	1690.28	1950.07	9.25 o
1	2	0	8141.63	8183.52	39.44 o
2	2	0	100486.86	104603.63	256.92 o
3	2	0	5773.87	6280.70	27.61 o
4	2	0	1056.10	984.18	7.36 o
5	2	0	130.06	132.16	5.25 o
6	2	0	242.72	192.46	10.42 o
7	2	0	389.14	416.18	15.63 o
8	2	0	423.40	389.86	16.18 o
-8	3	0	73.08	59.49	18.09 o
-7	3	0	94.16	69.91	13.98 o
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-5	3	0	1353.65	1336.85	23.44 o
-4	3	0	721.47	590.32	7.28 o
-3	3	0	103.15	85.35	4.32 o
-2	3	0	6.09	21.06	3.75 o
-1	3	0	3240.41	3579.10	20.52 o
0	3	0	2692.22	2791.76	15.22 o
1	3	0	848.21	1050.25	12.73 o
2	3	0	862.43	966.94	10.83 o
3	3	0	636.14	622.65	9.87 o
4	3	0	491.35	494.47	8.95 o
5	3	0	835.32	838.25	13.71 o
6	3	0	701.95	648.40	15.35 o
7	3	0	389.79	395.62	13.16 o
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-8	4	0	41.94	37.83	18.09 o
-7	4	0	6.64	23.60	10.39 o
-6	4	0	301.68	249.84	10.92 o
-5	4	0	292.79	357.53	11.14 o

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-2	4	0	9592.48	9769.56	33.29	o
-1	4	0	5958.45	6608.57	23.53	o
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3	4	0	7653.14	8400.24	30.38	o
4	4	0	15698.55	16061.92	70.07	o
5	4	0	311.68	365.02	13.43	o
6	4	0	175.95	142.57	11.79	o
7	4	0	6.41	1.64	11.51	o
8	4	0	60.30	73.75	15.08	o
-8	5	0	168.13	200.14	21.11	o
-7	5	0	0.90	6.37	11.46	o
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-3	5	0	1114.93	1043.71	11.30	o
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2	5	0	4632.05	4796.81	22.67	o
3	5	0	358.20	362.91	6.12	o
4	5	0	37.03	30.52	8.52	o
5	5	0	302.19	281.26	11.05	o
6	5	0	259.05	272.16	11.24	o
7	5	0	518.60	481.71	19.74	o
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-5	6	0	4.42	9.42	8.81	o
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-3	6	0	652.83	735.70	10.66	o
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2	6	0	3006.76	3011.41	21.25	o
3	6	0	397.45	407.45	7.74	o
4	6	0	170.13	190.75	9.78	o
5	6	0	2799.97	2824.14	32.21	o
6	6	0	4361.69	4268.76	34.31	o
7	6	0	79.45	77.04	17.55	o
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3	7	0	295.19	299.18	12.78 o
4	7	0	515.34	471.40	13.06 o
5	7	0	508.23	587.20	14.83 o
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7	7	0	112.66	90.20	17.55 o
-7	8	0	39.44	41.21	13.75 o
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3	8	0	379.24	360.11	8.90 o
4	8	0	277.72	270.44	13.57 o
5	8	0	21.92	13.91	9.59 o
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-3	9	0	70.36	49.04	7.66 o
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-1	9	0	6.44	19.31	7.02 o
0	9	0	2012.26	1928.38	36.60 o
1	9	0	993.91	960.95	12.69 o
2	9	0	726.65	745.61	12.13 o
3	9	0	10.01	15.38	7.08 o
4	9	0	2.05	3.39	9.75 o
5	9	0	224.68	231.66	13.86 o
6	9	0	85.49	87.12	12.49 o
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-5	10	0	62.53	94.60	14.03 o
-4	10	0	81.94	52.36	9.70 o
-3	10	0	263.19	258.02	11.93 o
-2	10	0	25.40	18.73	8.47 o
-1	10	0	138.86	150.63	8.67 o
0	10	0	1259.68	1273.66	16.96 o

```
 1  10   0     263.25     262.16     10.97 o
 2  10   0       8.36      10.48      7.67 o
 3  10   0    1002.61     974.11     14.57 o
 4  10   0     708.13     708.04     19.77 o
 5  10   0     245.53     258.45     14.82 o
 6  10   0       3.33      10.10     13.34 o
-5  11   0      35.22      15.63     18.64 o
-4  11   0     220.61     241.43     13.39 o
-3  11   0     252.93     241.99     12.80 o
-2  11   0     359.82     387.37     13.39 o
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 2  11   0     429.13     478.67     13.69 o
 3  11   0     439.30     437.70     13.10 o
 4  11   0     620.03     614.36     19.58 o
 5  11   0      35.27      17.72     13.85 o
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-3  12   0      21.98      42.39     11.16 o
-2  12   0      96.52      78.37     11.14 o
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-2  14   0     101.80      86.65     13.89 o
-1  14   0      58.47      49.99     15.59 o
 0  14   0      51.27      45.63     13.13 o
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 2  14   0       1.26      10.35     12.84 o
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-2 -14   1     103.56      91.76     18.50 o
-1 -14   1     130.09      85.65     14.47 o
 0 -14   1     159.23     164.74     15.74 o
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 2 -14   1      50.85      60.32     26.32 o
-3 -13   1     105.64     111.10     14.82 o
-2 -13   1       5.33       0.27     12.59 o
```

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 0 -13   1      64.13      48.94     12.06 o
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 2 -13   1       3.06       6.28     11.93 o
 3 -13   1     170.49     181.67     15.10 o
 4 -13   1     100.17      73.63     18.11 o
-4 -12   1     100.24     125.57     23.30 o
-3 -12   1     220.41     227.49     14.46 o
-2 -12   1      61.40      58.93     11.73 o
-1 -12   1      28.10      22.48     10.84 o
 0 -12   1      89.21      78.22     17.75 o
 1 -12   1     433.59     412.80     13.23 o
 2 -12   1     413.21     425.50     16.33 o
 3 -12   1      64.81      75.91     12.07 o
 4 -12   1       5.64       3.45     12.20 o
-5 -11   1     184.30     178.69     17.05 o
-4 -11   1     200.68     212.48     23.44 o
-3 -11   1     178.41     204.60     12.90 o
-2 -11   1      87.38      93.83     10.97 o
-1 -11   1      33.93      24.54     10.08 o
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 2 -11   1      53.00      54.17     10.56 o
 3 -11   1      27.68      20.33     10.26 o
 4 -11   1       0.60       7.46     10.74 o
 5 -11   1     289.25     373.69     27.14 o
-6 -10   1      19.25      13.03     14.22 o
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-4 -10   1       3.53       4.25     12.02 o
-3 -10   1       1.43       3.06      8.08 o
-2 -10   1      92.20      90.89     10.34 o
-1 -10   1     165.69     156.03      9.91 o
 0 -10   1     260.98     286.15     10.86 o
 1 -10   1     142.53     123.31      9.36 o
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 3 -10   1     320.73     289.85     12.59 o
 4 -10   1     567.82     539.46     23.22 o
 5 -10   1     213.45     257.62     18.92 o
 6 -10   1       0.68       4.05     14.40 o
-6  -9   1       8.27       0.00     12.21 o
-5  -9   1      27.92      16.28     11.75 o
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 3  -9   1    1550.28    1611.25     18.67 o
```

```
 4  -9   1      74.77      53.32      8.78 o
 5  -9   1      49.34      32.97     12.00 o
 6  -9   1       0.12       4.66     12.80 o
 7  -9   1     122.88     116.15     22.89 o
-7  -8   1     191.59     138.18     20.01 o
-6  -8   1      17.52       8.47     11.33 o
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-4  -8   1     390.04     382.78     15.76 o
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 6  -8   1     142.05      91.32     12.70 o
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-7  -7   1     937.86     856.49     28.51 o
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-4  -7   1      95.14      87.78     10.45 o
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 4  -7   1       7.85       9.54      6.94 o
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5	-8	2	688.05	702.13	19.76	o
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-6  -8   4      78.20      76.67     16.28 o
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 5  -9   5     94.08     105.01     24.95 o
 6  -9   5    161.17     168.34     28.79 o
-5  -8   5      3.15       6.78     15.39 o
-4  -8   5    332.26     311.68     18.11 o
-3  -8   5    291.84     295.62     13.87 o
-2  -8   5     16.61      11.09     10.34 o
-1  -8   5    449.46     455.66     14.72 o
 0  -8   5    538.16     623.17     13.28 o
 1  -8   5     48.70      67.07      9.78 o
 2  -8   5    153.51     146.26     11.69 o
 3  -8   5    159.55     146.23     11.23 o
 4  -8   5    369.83     337.44     14.76 o
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 5  -8   5      54.13      50.87     12.51 o
 6  -8   5      59.58      70.71     14.65 o
-6  -7   5       2.10       4.95     16.15 o
-5  -7   5       0.89       0.00     13.97 o
-4  -7   5     170.03     200.03     15.67 o
-3  -7   5       6.69       4.65      8.90 o
-2  -7   5      96.62     101.57     10.49 o
-1  -7   5      51.50      50.05     10.33 o
 0  -7   5     303.12     308.28     16.54 o
 1  -7   5      68.58      71.40      7.74 o
 2  -7   5     286.07     254.33     11.85 o
 3  -7   5      33.19      25.84      7.00 o
 4  -7   5     342.35     298.13      8.90 o
 5  -7   5      45.55      59.68     10.69 o
 6  -7   5      43.57      12.08     12.25 o
 7  -7   5      20.08      22.61     14.75 o
-6  -6   5      26.15      20.81     16.28 o
-5  -6   5     254.60     266.76     18.23 o
-4  -6   5     143.69     140.37     14.34 o
-3  -6   5       0.14       0.82      8.01 o
-2  -6   5     625.00     605.77     15.31 o
-1  -6   5     832.09     769.91     24.98 o
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 3  -6   5      68.63      77.17      6.76 o
 4  -6   5     371.25     397.11      9.63 o
 5  -6   5     239.57     300.22     12.45 o
 6  -6   5      77.39      48.08     14.84 o
 7  -6   5      28.27      36.70     13.19 o
-6  -5   5     186.71     149.23     18.09 o
-5  -5   5      11.25      13.91     12.50 o
-4  -5   5       0.32       0.54     11.70 o
-3  -5   5     101.65      88.85      7.64 o
-2  -5   5      72.68      41.66      7.18 o
-1  -5   5       4.09       5.68      5.10 o
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 5  -5   5      58.95      87.88      8.90 o
 6  -5   5     176.11     148.12     10.55 o
 7  -5   5      42.45      45.08     12.34 o
-7  -4   5      12.26      16.67     15.95 o
-6  -4   5       0.21       9.10     12.86 o
-5  -4   5     426.71     385.53     17.82 o
-4  -4   5      92.75     115.48      9.55 o
-3  -4   5     202.17     185.50      8.57 o
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5	-4	5	62.59	27.70	8.58 o
6	-4	5	204.50	189.11	9.91 o
7	-4	5	253.48	383.98	14.01 o
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6	2	5	19.82	2.47	18.92 o
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2	14	5	52.33	60.17	18.51 o
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-6   4   9    295.49     326.07    18.15 o
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3	11	10	840.65	829.10	14.17	o
4	11	10	82.17	95.54	13.97	o
5	11	10	48.18	47.76	13.10	o
6	11	10	4.48	5.86	14.40	o
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-3	12	10	405.16	495.47	27.96	o

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-2  12  10     192.26     172.91     13.18 o
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 4  12  10     241.43     238.87     20.15 o
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 3  13  10     227.67     246.63     11.99 o
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-2  14  10     202.85     200.89     15.27 o
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 1  14  10       4.41      12.78     11.33 o
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 3  14  10       6.71       9.47     11.42 o
 4  14  10     169.89     188.95     18.64 o
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 4  15  10      11.33       1.64     23.30 o
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 3   5  12     130.74    114.42     9.45 o
 4   5  12       8.16      8.42    12.12 o
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 5   9  14     489.95     460.29     19.86 o
 6   9  14      17.93      15.64     16.04 o
```

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2	10	14	207.09	191.55	13.92	o
3	10	14	319.62	314.93	15.29	o
4	10	14	509.76	452.92	26.59	o
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3	11	14	162.25	148.24	13.53	o
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5	11	14	157.03	174.16	16.85	o
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2	12	14	18.64	5.98	12.19	o
3	12	14	141.84	114.01	13.51	o
4	12	14	177.03	189.45	17.63	o
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1	13	14	3.17	29.99	13.03	o
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3	13	14	436.58	456.70	18.14	o
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0	14	14	297.22	363.82	31.80	o
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 4   3  15       1.72       0.27     20.84 o
 5   3  15       0.23       2.80     14.81 o
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-2   7  15      15.53      21.20     14.02 o
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 4   9  15     162.88     163.77     18.67 o
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 0   4  16      66.83      46.80     17.40 o
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 1   5  16     100.46     149.94     15.71 o
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 5   6  16     139.59     165.05     27.42 o
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 3   7  16     113.10     125.72     15.19 o
 4   7  16     232.18     266.76     30.16 o
 5   7  16     222.63     187.26     30.43 o
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 3   8  16      34.99      33.17     14.72 o
 4   8  16      37.77      54.83     25.77 o
 5   8  16       8.67      19.74     28.24 o
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 2   6  17      26.33      23.22     15.42 o
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 2   7  17     124.88     113.06     15.71 o
 3   7  17       2.82       0.27     17.59 o
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   1   9  17       25.74       17.27     30.71 o
   2   9  17       44.81       48.10     16.63 o
   3   9  17      104.49      102.02     21.21 o
   2  10  17       10.89        0.00     31.25 o

===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
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data_[Ni(tpt)2](ClO4)2, 2.13 (E)
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_shelx_F_calc_maximum      297.47
_exptl_crystal_F_000      1800.00
_reflns_d_resolution_high  0.7876

loop_
 _symmetry_equiv_pos_as_xyz
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 '-x, -y, z+1/2'
 'x+1/2, -y, z'
 '-x+1/2, y, z+1/2'

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_cell_length_c    21.6363
_cell_angle_alpha  90.000
_cell_angle_beta   90.000
_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
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 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
   4   0 -26     1038.81     1314.86    191.57 o
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   4   1 -26      518.95      278.65    149.53 o
   3   2 -26       72.55      -66.74    114.01 o
   4   2 -26      321.33      159.30    195.64 o
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2   0 -25    217.15     481.49    118.20 o
4   0 -25     17.60     157.45     91.92 o
6   0 -25    735.01     777.14    109.66 o
1   1 -25    219.41     150.35     74.25 o
2   1 -25   1010.37     984.71     85.35 o
3   1 -25     29.70      36.61     60.07 o
4   1 -25     64.63      59.43     61.24 o
5   1 -25    461.58     558.94     65.91 o
6   1 -25    151.01     203.45    126.52 o
1   2 -25     64.80     124.25     70.87 o
2   2 -25    119.59      62.87    103.77 o
3   2 -25     23.50      24.85     57.27 o
4   2 -25    745.68     604.17     76.54 o
5   2 -25    103.87     151.13     75.49 o
6   2 -25    161.13      85.61     89.24 o
1   3 -25     38.45     126.40     69.80 o
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3   3 -25    124.76      60.38     77.96 o
4   3 -25    211.49      77.08     78.22 o
5   3 -25     63.57      64.96     83.74 o
6   3 -25     54.53      45.78    131.17 o
3   4 -25    281.90      84.88    120.77 o
4   4 -25    401.52     114.82    188.98 o
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4   0 -24     21.38     -45.74     84.45 o
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9   2 -22      94.55      94.61     90.03 o
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6   3 -22    1558.31    1420.13     68.33 o
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 9   9  -2     868.59     843.93     78.56 o
10   9  -2     542.65     511.38     57.40 o
11   9  -2     266.72     269.70     55.56 o
12   9  -2     129.88     143.56     58.85 o
13   9  -2     152.10      70.39     71.73 o
14   9  -2       6.22       6.80     66.14 o
15   9  -2      66.41      -8.17    114.52 o
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 1  10  -2     332.54     479.08     49.86 o
 2  10  -2     495.16     503.04     55.47 o
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 4  10  -2     975.78     971.02     67.47 o
 5  10  -2     785.85     728.68     65.34 o
 6  10  -2      54.57      64.64     52.29 o
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11  10  -2      53.47     186.19     69.05 o
12  10  -2      68.83      72.59     69.69 o
13  10  -2      14.27      18.71     74.27 o
14  10  -2     265.53     331.36    151.88 o
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3	6	0	20.12	35.94	26.29	o
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5	6	0	240.02	262.64	30.23	o
6	6	0	1023.61	1000.69	43.80	o
7	6	0	5147.93	5130.86	61.66	o
8	6	0	33.88	-33.43	33.43	o
9	6	0	33.97	22.11	37.14	o
10	6	0	2083.27	2148.95	73.89	o
11	6	0	339.71	301.48	43.42	o
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13	6	0	29.13	9.47	52.11	o
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16	6	0	3.21	93.14	88.78	o
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4	8	0	376.09	417.93	42.94	o
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 4  10   0    1773.56    1866.30     76.94 o
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9	3	2	814.12	805.35	25.97	o
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16	3	2	2926.53	3097.79	107.96	o
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19	3	2	158.98	101.33	191.32	o
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 10   0   4    9538.97    9090.83    206.36 o
 12   0   4   16373.86   16275.33    297.59 o
 14   0   4     342.34     149.16    124.02 o
  0   1   4   12258.50   11855.46     64.42 o
  1   1   4      31.34       9.36     12.65 o
  2   1   4   84985.61   83370.82    205.04 o
  3   1   4   14847.50   15726.46    111.02 o
  4   1   4    7711.56    7441.21     56.54 o
  5   1   4    2236.80    2310.18     21.39 o
  6   1   4    6893.39    6847.78     53.94 o
  7   1   4     301.00     304.14     18.15 o
  8   1   4    5287.60    5457.06     61.43 o
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 11   1   4    1157.49    1356.77     83.59 o
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 13   1   4     110.93     168.64    114.15 o
 14   1   4    2408.91    2538.74    164.94 o
 15   1   4      71.31     134.62    129.69 o
 16   1   4     184.88      60.65    139.79 o
  0   2   4   88485.54   85568.80    357.50 o
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5	2	4	773.02	804.29	21.42 o
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7	2	4	310.44	325.87	17.39 o
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9	2	4	395.74	427.38	20.97 o
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5	3	4	691.85	728.45	22.81 o
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 7  12   5     68.51     129.53    77.13 o
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 2  13   5     26.16     104.03    79.17 o
 3  13   5    268.22     337.61   147.68 o
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 3  11   7     160.18     136.32     67.36 o
 4  11   7     611.63     700.84     74.69 o
 5  11   7     365.63     366.81     81.66 o
 6  11   7      24.80      97.72     68.68 o
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 9  11   7      42.56      59.99     77.04 o
10  11   7     139.40     123.71     86.03 o
12  11   7       1.83      29.34    208.09 o
 1  12   7     219.50     195.42     73.63 o
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 3  12   7     356.82     458.14     79.70 o
 4  12   7     292.22     284.47     93.63 o
 5  12   7     353.40     433.52     81.65 o
 6  12   7      25.42      23.95     76.21 o
 7  12   7     130.54     213.75     81.61 o
 8  12   7     247.47     196.99    160.63 o
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 2  13   7      79.03     177.52     84.24 o
 3  13   7      60.07     123.44     84.25 o
 4  13   7     214.72     342.07     86.05 o
 5  13   7     377.52     205.18    106.01 o
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 4   0   8    7389.85    7018.52     68.67 o
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 5   1   8     708.40     785.94     22.08 o
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14	9	9	440.85	387.33	153.85	o
1	10	9	711.79	736.69	56.57	o
2	10	9	745.32	775.38	63.11	o

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 4  10   9     249.35     166.65     92.84 o
 5  10   9    1479.54    1437.52     81.73 o
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10  10   9     738.99     664.13     79.21 o
11  10   9     358.22     383.93     77.86 o
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 3  11   9     144.57     168.14     71.13 o
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 5  11   9     261.01     244.82     72.47 o
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 7  11   9     392.00     370.41     82.12 o
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 9  11   9     193.06     140.44    106.26 o
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11  11   9     392.06     276.63    158.78 o
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6	10	11	268.20	126.28	68.27	o
7	10	11	309.77	266.69	82.24	o
8	10	11	230.01	114.21	61.57	o
9	10	11	80.32	83.10	67.87	o
10	10	11	206.10	190.20	89.93	o
11	10	11	106.69	66.83	116.87	o
1	11	11	653.86	633.03	69.22	o
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4	11	11	375.56	344.65	76.60	o
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7	11	11	129.76	178.87	78.54	o
8	11	11	302.11	298.11	80.30	o

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 0   2  14     465.39     529.66     58.43 o
 1   2  14     209.42     211.89     24.95 o
 2   2  14     888.04     931.87     30.73 o
 3   2  14     520.24     466.54     27.54 o
 4   2  14    5077.45    4725.94     52.01 o
 5   2  14     223.69     235.28     31.33 o
 6   2  14    5335.66    5222.44     76.75 o
 7   2  14      61.04       1.52     29.88 o
 8   2  14    2053.30    2053.99     59.01 o
 9   2  14       4.44      16.60     35.70 o
10   2  14    1880.03    1881.47     79.96 o
11   2  14      13.29       8.05     46.01 o
12   2  14     275.87     260.80     50.12 o
13   2  14      45.93      41.98     48.61 o
14   2  14     389.81     359.41     59.98 o
15   2  14     191.82     314.54    123.52 o
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6	5	14	3302.13	3409.88	72.15	o
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4	8	14	1746.74	1853.20	66.31	o
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3	9	14	1523.61	1570.84	66.67	o
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6	10	14	404.54	471.02	61.44	o
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12	1	15	473.50	459.82	53.44	o
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 9   2  15      80.36     89.29     38.43 o
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11   2  15     426.25    363.66     52.26 o
12   2  15     835.10    782.08     58.31 o
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 5   3  15      47.66     68.08     34.21 o
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10   5  15    1575.21    1552.59     93.18 o
11   5  15     214.84     221.30     56.30 o
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13   5  15     167.41     153.01     64.42 o
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11   6  16      35.74      28.41     63.24 o
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13   6  16     256.06      99.77     83.78 o
 0   7  16     533.00     690.58     65.69 o
 1   7  16     138.63     141.86     41.16 o
 2   7  16     377.12     387.51     43.73 o
 3   7  16     519.74     584.90     53.09 o
 4   7  16    1161.89    1182.53     59.99 o
 5   7  16     187.86     132.43     49.81 o
 6   7  16    1820.03    1651.91     66.35 o
 7   7  16     511.08     432.92     50.62 o
 8   7  16     231.06     219.86     58.09 o
 9   7  16     109.43     114.42     54.54 o
10   7  16     489.92     490.34     73.13 o
11   7  16      16.34      -4.53     70.22 o
12   7  16     416.50     324.17     85.84 o
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 1   8  16     289.47     244.60     44.99 o
 2   8  16      55.25      26.21     46.24 o
 3   8  16    1380.23    1343.28     63.40 o
 4   8  16     456.56     542.89     79.62 o
 5   8  16      81.56      67.12     51.85 o
 6   8  16     624.65     646.23     61.24 o
 7   8  16     314.88     394.54     59.29 o
 8   8  16      51.29      24.37     71.93 o
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 3   9  16    1248.87    1362.86     70.30 o
 4   9  16      30.94      -6.07     55.34 o
 5   9  16      45.80      62.44     57.81 o
 6   9  16     357.00     421.00     62.53 o
 7   9  16     219.99     109.11     67.21 o
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 5  10  16     324.06     313.87     64.53 o
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 8  10  16     187.13     134.37    175.79 o
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 6   1  17     230.12     302.22     36.20 o
 7   1  17     133.75     101.49     35.61 o
 8   1  17    1321.58    1408.90     53.50 o
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10   1  17    1134.94    1095.94     63.59 o
11   1  17      41.44      48.15     52.00 o
12   1  17     301.41     336.19     57.61 o
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14   1  17     496.53     437.74     64.06 o
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6	4	17	64.57	89.61	37.14	o
7	4	17	145.84	185.05	39.72	o
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8	5	17	2045.81	2126.52	67.19	o

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10   6  17     185.45     277.89     65.41 o
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11   7  17     420.30     445.31     87.98 o
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 2   0  18    2346.63    2460.65     77.40 o
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 6   0  18    4633.40    4613.19     97.79 o
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12   0  18     175.74     112.10     82.42 o
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 6   2  18    3051.62    2949.71     57.31 o
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5	6	19	543.69	512.80	63.69 o
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7	6	19	92.83	126.11	54.38 o
8	6	19	966.75	1110.47	76.67 o
9	6	19	95.36	107.47	60.24 o
10	6	19	429.62	391.40	87.04 o
11	6	19	41.08	-13.03	108.71 o
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2	7	19	1051.99	1094.44	63.54 o
3	7	19	31.83	-52.74	52.74 o
4	7	19	721.08	799.12	63.26 o
5	7	19	253.42	347.75	59.50 o
6	7	19	105.12	94.75	57.87 o
7	7	19	99.80	168.74	65.94 o
8	7	19	252.81	287.62	73.82 o
9	7	19	97.24	317.02	116.96 o
10	7	19	168.02	-147.93	147.93 o
1	8	19	479.83	462.02	61.98 o
2	8	19	629.82	821.73	66.52 o
3	8	19	62.18	2.58	59.48 o
4	8	19	634.75	683.74	65.77 o
5	8	19	359.44	352.33	63.24 o
6	8	19	113.68	111.43	62.94 o
7	8	19	313.95	188.32	84.90 o
8	8	19	399.88	406.91	125.35 o
9	8	19	122.23	-139.30	199.21 o
1	9	19	592.14	530.47	67.75 o
2	9	19	753.23	865.22	71.90 o
3	9	19	18.30	0.88	63.93 o
4	9	19	114.92	165.88	66.55 o

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3  4  24       0.51     111.92     83.12 o
4  4  24     108.61     203.52     69.08 o
5  4  24      73.53     128.50     81.68 o
6  4  24     609.14     551.35    135.60 o
0  5  24    1808.78    1812.23    127.68 o
1  5  24      51.70      37.08     89.75 o
2  5  24     198.64     183.71    115.09 o
3  5  24      55.34      23.49     74.01 o
4  5  24     136.57     -53.63    100.56 o
5  5  24       1.44      39.26    232.74 o
6  5  24     139.45       5.62    283.53 o
0  6  24     679.01     521.46    163.71 o
1  6  24     119.95     176.63    119.34 o
2  6  24     135.99     222.55    117.46 o
0  7  24     838.75     510.85    173.08 o
2  0  25     234.20     150.38     79.15 o
4  0  25      23.72     -59.28     76.04 o
6  0  25     752.36     561.15    162.97 o
1  1  25     211.51     241.22     52.50 o
2  1  25    1008.90    1183.55    112.00 o
3  1  25      30.21      58.74     54.71 o
4  1  25      82.08     114.34     65.90 o
5  1  25     449.92     402.25     61.14 o
6  1  25     148.79     186.30    126.48 o
```

```
   1   2  25       71.90      160.04     54.32 o
   2   2  25      164.40      119.93     68.91 o
   3   2  25       22.24       19.53     57.50 o
   4   2  25      741.23      662.06     65.96 o
   5   2  25       97.11      271.30     89.99 o
   6   2  25      152.76      228.31    190.83 o
   1   3  25       28.87      113.12     59.99 o
   2   3  25     1039.89     1031.28     77.57 o
   3   3  25      128.70      255.73     69.39 o
   4   3  25      223.37      246.45     80.40 o
   5   3  25       51.13      345.35    120.21 o
   6   3  25       67.42      419.59    137.06 o
   1   4  25      252.90      468.41    121.95 o
   2   4  25      298.75      422.37    119.02 o
   3   4  25      287.23      345.00     90.85 o
   4   4  25      357.31      325.94    164.70 o
   5   4  25      153.73      339.75    166.92 o
   1   5  25      109.01       20.16    150.77 o
   2   5  25     1147.72     1018.01    210.80 o
   0   0  26      478.33      336.30    143.99 o
   2   0  26      238.25      330.47    139.05 o
   4   0  26     1171.82     1232.51    171.11 o
   0   1  26     1172.92      756.78    219.92 o
   1   1  26        5.97       33.21     83.73 o
   2   1  26       37.87       22.04     85.32 o
   3   1  26      136.38      154.52     73.78 o
   4   1  26      556.14      522.14    117.33 o
   5   1  26      110.22      210.13    122.17 o
   0   2  26       62.40      105.03    111.02 o
   1   2  26       21.50       39.40    111.67 o
   2   2  26       13.36      -12.90    109.40 o
   0   3  26      536.06      353.67    127.54 o
   1   3  26       87.74      123.31    113.72 o
   2   3  26       18.05       29.83    177.02 o
   0   4  26      229.16      210.80    165.19 o
   1   4  26       75.09      102.81    202.91 o
   1   1  27       30.95      -73.74    109.48 o

===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_[Ni2(tpt)(EtOH)2(NO3)3(H2O)](NO3), 2.15 (F)
_shelx_title ' 2.15 (F) in P-1'
_shelx_refln_list_code          4
_shelx_F_calc_maximum      228.69
_exptl_crystal_F_000       808.00
_reflns_d_resolution_high  0.7991
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 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, -y, -z'

_cell_length_a     7.3769
_cell_length_b    14.0212
_cell_length_c    15.1672
_cell_angle_alpha  73.018
_cell_angle_beta   84.192
_cell_angle_gamma  89.706

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
   1   0   0    2611.49    2418.26     96.57 o
   2   0   0    7544.07    7902.20    273.54 o
   3   0   0    6662.38    6508.18    232.55 o
   4   0   0       2.14       3.08     31.98 o
   5   0   0     280.96     359.61     35.84 o
   6   0   0     423.34     417.22     30.66 o
   9   0   0       6.82     -14.43    116.00 o
  -7   1   0    1385.07    1447.22     94.07 o
  -6   1   0     739.99     785.55     42.58 o
  -5   1   0     521.23     551.15     32.04 o
  -4   1   0    1302.32    1328.91     87.96 o
  -3   1   0      74.16      38.20     14.90 o
  -2   1   0   52300.23   54083.37   1858.84 o
  -1   1   0      45.59      64.81      7.65 o
   1   1   0    6011.22    6040.71    368.53 o
   2   1   0       0.83     -10.18     10.18 o
   3   1   0     362.94     445.54     58.55 o
   4   1   0    6198.90    6086.22    188.94 o
   5   1   0    2896.06    2782.35     90.58 o
   6   1   0     341.43     299.37     43.85 o
   8   1   0     116.80     160.40     92.69 o
   9   1   0      69.90      80.48    108.78 o
  -7   2   0     415.31     397.14     39.25 o
  -6   2   0     569.77     678.32     65.21 o
  -5   2   0      19.73       5.01     21.07 o
  -4   2   0    9653.19   10326.07    317.17 o
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 3   2   0     691.88     587.75     36.08 o
 4   2   0    4915.89    4898.38    136.02 o
 5   2   0     145.87     121.05     22.29 o
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 7   2   0     891.86     862.23     67.50 o
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 9   2   0     162.07     202.58    107.12 o
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 1   3   0    6011.40    6347.61    174.73 o
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 3   3   0    2449.22    2555.85     71.71 o
 4   3   0    1416.19    1519.22     80.75 o
 5   3   0    4091.53    4322.64    201.83 o
 6   3   0      70.05      96.74     43.20 o
 7   3   0     752.31     710.89     81.86 o
 8   3   0    2961.34    2850.52    206.46 o
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-7   4   0    1289.65    1285.54     64.56 o
-6   4   0    2288.84    2206.65    170.94 o
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-3   4   0     183.43     226.17     36.35 o
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 2   4   0      83.84      82.99     18.99 o
 3   4   0    1609.33    1597.98     72.42 o
 4   4   0     944.89    1029.25     72.01 o
 5   4   0     254.76     275.11     37.68 o
 6   4   0    2044.45    2168.64    200.36 o
 7   4   0      39.85     -12.61     46.23 o
 8   4   0     109.98      80.93     57.89 o
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3	5	0	2473.31	2281.19	78.64	o
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3	6	0	2573.72	2717.99	180.38	o
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 3 -14   1     851.09     828.78     54.48 o
 4 -14   1      66.51      44.72     41.79 o
 5 -14   1     502.51     550.63     54.44 o
-5 -13   1     638.41     857.49    109.34 o
-4 -13   1     785.21     951.28    106.56 o
-3 -13   1       4.27     127.10     67.16 o
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 4 -13   1      13.78       0.98     37.04 o
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-4 -10   1     171.92     229.22     61.61 o
-3 -10   1    2306.52    2532.50    187.04 o
-2 -10   1     109.28     142.64     51.62 o
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 5 -10   1      20.36       8.05     38.85 o
 6 -10   1       3.87       9.41     37.47 o
 7 -10   1     110.86     145.01     45.87 o
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 3  10   3      22.96      53.28     54.39 o
 4  10   3     199.09     139.95     40.54 o
 5  10   3    1161.11    1116.10    130.15 o
 6  10   3     567.55     588.65    142.91 o
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-6  11   3      24.12      42.65     44.04 o
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-7   9   4     533.69     599.05     53.15 o
-6   9   4       2.92      16.13     36.05 o
-5   9   4     265.12     260.11     35.91 o
-4   9   4    5684.68    5792.20    208.58 o
-3   9   4     197.18     172.14     28.80 o
-2   9   4     811.33     851.58     41.20 o
-1   9   4    1192.93    1013.65     44.14 o
 0   9   4     134.32     145.50     23.13 o
 1   9   4    1954.32    1682.79    125.99 o
 2   9   4    1777.10    1899.24    143.19 o
 3   9   4     148.63     101.79     32.84 o
 4   9   4    1330.79    1344.77     76.33 o
 5   9   4    1221.80    1160.12     72.84 o
 6   9   4     493.39     437.09     82.14 o
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 5  10   4    1106.97    1062.65     69.34 o
 6  10   4    1212.15    1291.94     82.13 o
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-7  11   4     949.97     994.39     69.07 o
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-6  12   4      41.47      -6.91     47.02 o
-5  12   4     783.71     769.07     52.94 o
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-4   2  15      285.63      297.17     43.37 o
-3   2  15     1693.63     1608.58     96.29 o
-2   2  15      102.02      107.51     34.60 o
-1   2  15      290.77      269.37     35.65 o
 0   2  15     2199.94     2370.09     95.56 o
 1   2  15        0.20       56.13     31.68 o
 2   2  15        5.34       39.27     32.27 o
 3   2  15      941.16      994.04    110.72 o
-4   3  15      630.14      597.17     49.82 o
-3   3  15       49.09       61.72     35.71 o
-2   3  15      161.83      180.75     36.79 o
-1   3  15      151.20      122.89     32.12 o
 0   3  15      289.74      273.42     35.15 o
 1   3  15     1051.81     1157.01     57.44 o
 2   3  15      116.10      155.41     32.52 o
 3   3  15       73.09       33.44     33.96 o
-4   4  15       92.45       83.53     39.72 o
-3   4  15     1812.75     1630.78     75.12 o
-2   4  15      663.47      657.04     46.11 o
-1   4  15       93.31      124.61     32.40 o
 0   4  15     1697.56     1839.99    177.05 o
 1   4  15      203.06      281.14     50.51 o
 2   4  15       15.84       -3.16     32.87 o
-4   5  15        2.44       -2.66     37.83 o
-3   5  15     1865.93     1857.82    186.21 o
-2   5  15       15.74       22.64     33.25 o
-1   5  15      313.64      351.43     54.39 o
 0   5  15       68.88      152.11     54.95 o
 1   5  15     1117.99     1134.17     56.05 o
 2   5  15      589.67      661.02     66.60 o
-4   6  15      248.35      188.02     38.70 o
-3   6  15       74.48       63.11     35.87 o
-2   6  15     1309.23     1242.30     61.19 o
-1   6  15      241.90      201.40     35.44 o
 0   6  15      211.98      164.55     33.19 o
 1   6  15     1304.71     1293.34     60.59 o
-4   7  15        5.35      -16.86     59.66 o
-3   7  15      152.89       80.19     49.67 o
-2   7  15       89.76      106.31     35.95 o
-1   7  15      253.72      340.34     37.85 o
 0   7  15      651.50      656.14    125.71 o
-4   8  15      236.46      274.14     41.74 o
-3   8  15       79.40      109.11     37.22 o
```

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-2   8  15      998.94     1004.29     54.14 o
-1   8  15      847.00      820.70     49.05 o
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-4   9  15      124.25      104.31     41.70 o
-3   9  15      608.10      603.41     49.73 o
-2   9  15       82.17      108.44     35.79 o
-1   9  15      103.41      106.06     33.69 o
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-2  10  15        1.35       -4.00     35.56 o
-1  10  15     1200.82     1065.06     84.36 o
-3  11  15      926.48      972.19     62.91 o
-2  11  15      134.36      111.74     40.11 o
 0  -4  16       14.38       50.25     44.25 o
 1  -4  16      665.69      791.95     59.51 o
 2  -4  16      106.67       84.82     47.33 o
-1  -3  16      328.71      306.38     47.07 o
 0  -3  16      436.79      546.79     50.01 o
 1  -3  16       16.94       47.91     43.43 o
 2  -3  16      408.50      507.99     51.52 o
 3  -3  16      522.02      588.23     61.33 o
-2  -2  16       80.99       61.46     44.55 o
-1  -2  16     1067.23     1062.56    108.50 o
 0  -2  16      113.79      117.06     50.78 o
 1  -2  16        5.14       19.01     39.98 o
 2  -2  16      120.10      100.57     46.07 o
 3  -2  16       25.89      -10.06     40.90 o
 4  -2  16      364.97      398.16     55.22 o
-3  -1  16      417.57      525.48     92.96 o
-2  -1  16      759.60      813.17     56.71 o
-1  -1  16        0.68       12.00     38.98 o
 0  -1  16      510.52      596.19     48.89 o
 1  -1  16      537.91      566.00     48.29 o
 2  -1  16        0.65      -35.40     39.43 o
 3  -1  16     1139.22     1186.05     88.25 o
 4  -1  16      897.87      883.55    139.58 o
-3   0  16      352.92      393.84     48.85 o
-2   0  16      464.37      442.02     45.91 o
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 0   0  16      144.34      148.62     38.93 o
 1   0  16        1.25        3.83     35.59 o
 2   0  16       33.42       47.49     46.62 o
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 4   0  16       12.91      -16.60     54.39 o
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-2   1  16      711.51      739.70     50.79 o
-1   1  16      426.86      371.42     42.75 o
 0   1  16       57.59       58.62     35.61 o
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 2   1  16      324.54      341.03     39.98 o
```

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-1   2  16     178.60     185.32     42.74 o
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 3   2  16      93.22      73.47     38.36 o
-4   3  16     242.84     259.74     64.38 o
-3   3  16     281.16     192.93     41.37 o
-2   3  16     205.67     259.93     40.16 o
-1   3  16    3498.52    3285.07    188.98 o
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 1   3  16     529.14     621.66     44.87 o
 2   3  16    1602.47    1720.40     76.50 o
 3   3  16      44.99      27.75     58.28 o
-4   4  16     165.48     120.88     44.78 o
-3   4  16     240.74     300.58     67.16 o
-2   4  16    1228.73    1269.88     64.36 o
-1   4  16     353.16     445.28     42.25 o
 0   4  16     801.24     811.50     49.82 o
 1   4  16     162.52     132.08     35.87 o
 2   4  16       0.32      -4.95     35.72 o
-4   5  16     550.26     432.84     48.06 o
-3   5  16     301.83     229.46     41.16 o
-2   5  16     225.37     153.37     38.04 o
-1   5  16     954.06     959.58     54.33 o
 0   5  16    2070.21    2034.22     85.54 o
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-3   6  16      51.99      40.25     38.01 o
-2   6  16       4.76      19.09     36.67 o
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 0   6  16     434.73     470.87     41.51 o
 1   6  16     230.27     249.05     36.82 o
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-1   7  16     265.39     273.35     38.87 o
 0   7  16    1123.46    1167.38     60.04 o
-3   8  16       2.04     -12.99     39.17 o
-2   8  16     278.83     327.48     40.94 o
-1   8  16      96.67      79.35     37.85 o
 0   8  16       6.71      -3.33     53.84 o
-3   9  16     506.75     464.53     88.80 o
-2   9  16     880.40     915.38     55.05 o
-1   9  16     169.65      79.85     37.18 o
-3  10  16     211.91     291.87    129.04 o
-2  10  16     159.82     256.32     71.04 o
-2  11  16     499.71     512.42     50.10 o
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 2  -2  17     540.67     618.54     55.84 o
-1  -1  17      18.71      60.16     47.45 o
 0  -1  17      70.35     104.32     43.08 o
 1  -1  17     700.82     778.75    121.55 o
 2  -1  17       1.15     -24.69     42.58 o
 3  -1  17     178.22     253.54     57.17 o
-2   0  17      11.20     -41.58     41.58 o
-1   0  17     844.16     855.71     57.98 o
 0   0  17       3.05     -17.25     40.16 o
 1   0  17      13.53     -11.90     39.11 o
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 3   0  17     492.10     547.39     56.61 o
 4   0  17     135.53     131.98     48.36 o
-2   1  17     546.85     577.43     49.73 o
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 2   1  17     492.16     465.66     45.51 o
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-1   2  17      24.57      10.47     37.86 o
 0   2  17    1200.99    1324.91     67.81 o
 1   2  17     179.14     167.49     39.49 o
 2   2  17      57.26      37.94     38.52 o
 3   2  17    1594.13    1691.97    163.45 o
-3   3  17     216.00     291.59     56.33 o
-2   3  17      62.26      63.13     77.15 o
-1   3  17      56.25      62.08     89.36 o
 0   3  17     191.22     219.08     55.78 o
 1   3  17     243.71     238.60     41.00 o
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-3   4  17     328.94     373.93     47.97 o
-2   4  17      38.57      83.39     40.98 o
-1   4  17      57.28      97.68     39.10 o
 0   4  17     950.10    1001.96     99.90 o
 1   4  17     676.27     750.01     51.42 o
 2   4  17      21.19      37.60     38.48 o
-3   5  17     694.57     789.71     57.02 o
-2   5  17      11.81      34.54     49.95 o
-1   5  17      45.47      37.33     39.32 o
 0   5  17     696.67     727.95     49.90 o
 1   5  17     766.09     820.17     64.66 o
-3   6  17      16.41      22.23     42.34 o
-2   6  17     749.85     719.43     51.55 o
-1   6  17     121.01     134.60     49.40 o
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 1   6  17    1069.50    1239.33     91.58 o
```

```
  -3   7  17      149.83      213.19     43.30 o
  -2   7  17      865.19      838.96     96.02 o
  -1   7  17      243.84      333.23     45.79 o
   0   7  17      150.58      181.34     40.81 o
  -2   8  17        8.40       56.07     40.76 o
  -1   8  17      628.84      596.83     48.94 o
  -2   9  17      298.72      211.48     43.38 o
  -1   9  17        6.07       14.44     40.37 o
   0   0  18      604.05      515.05     75.48 o
   1   0  18       16.19       29.94     45.66 o
   2   0  18      223.64      295.82     74.37 o
  -1   1  18        0.71        6.11     66.60 o
   0   1  18       44.98       50.08     43.03 o
   1   1  18      637.45      690.54     55.62 o
   2   1  18      373.69      434.17     49.86 o
  -1   2  18       43.01       40.54     41.76 o
   0   2  18      156.47      179.21     48.29 o
   1   2  18      250.45      278.91     45.34 o
   2   2  18        7.33       76.76     45.28 o
   3   2  18       12.69        9.93     44.54 o
  -1   3  18      514.92      513.73     49.73 o
   0   3  18      121.93       72.67     92.13 o
   1   3  18      381.31      418.07     48.04 o
   2   3  18      219.71      228.62     46.01 o
  -1   4  18      218.07      186.80     43.87 o
   0   4  18       67.61      115.07     48.56 o
   1   4  18      828.39      904.65     81.86 o
   2   4  18        0.99       -6.11     66.05 o
  -1   5  18      383.48      389.68     49.95 o
   0   5  18      498.62      506.95     48.10 o
   1   5  18       22.07       37.90     41.51 o
  -1   6  18      335.51      399.53     48.64 o
   0   6  18       32.17       39.43     43.69 o
  -1   7  18        6.75       43.32     43.14 o
   0   7  18      870.65      800.12     70.49 o
  -1   8  18       21.51       79.24     45.79 o

===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_[Ru(tpm)(bpy)Cl](PF6), 3.10
_shelx_title ' 3.10 in P2(1)/n'
_shelx_refln_list_code          4
_shelx_F_calc_maximum      295.90
_exptl_crystal_F_000      1296.00
_reflns_d_resolution_high  0.7992
```

```
loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x+1/2, y+1/2, -z+1/2'
 '-x, -y, -z'
 'x-1/2, -y-1/2, z-1/2'

_cell_length_a    10.8239
_cell_length_b    15.0829
_cell_length_c    14.3141
_cell_angle_alpha  90.000
_cell_angle_beta   97.314
_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
  2   0   0    45804.34    45505.02    1319.90 o
  4   0   0     4530.26     3824.12     71.28 o
  6   0   0     5635.60     5331.47     99.63 o
  8   0   0     6468.64     6778.12    127.83 o
 10   0   0     7126.53     7333.99    146.94 o
 12   0   0     5281.78     5374.15    119.87 o
  1   1   0       28.32       10.03     11.86 o
  2   1   0     6846.40     5586.99    165.10 o
  3   1   0      315.75      180.77     13.23 o
  4   1   0      777.01      938.04     17.90 o
  5   1   0       94.05       77.80     20.78 o
  6   1   0      628.99      680.10     25.97 o
  7   1   0     2133.05     2173.44     35.57 o
  8   1   0      334.23      439.08     30.56 o
  9   1   0       97.55      144.37     37.22 o
 10   1   0     1334.29     1398.62     41.05 o
 11   1   0     1570.27     1533.11     65.44 o
 12   1   0     2454.61     2498.24     54.54 o
 13   1   0        0.02      -19.53     45.41 o
  0   2   0    18377.63    18011.55    523.58 o
  1   2   0    33402.76    31899.70    551.76 o
  2   2   0     8944.35     8822.25    235.90 o
  3   2   0     5338.46     5121.34     71.68 o
  4   2   0     5375.74     5330.63     69.13 o
  5   2   0     2617.06     2411.33     38.55 o
```

```
 6   2   0    1659.04    1639.92    28.64 o
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 9   2   0     869.99     839.01    37.80 o
10   2   0    2949.59    2988.44   102.05 o
11   2   0    4778.72    4967.40    77.84 o
12   2   0    1247.63    1273.79    45.66 o
13   2   0    1624.01    1629.61    52.48 o
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 2   3   0     540.69     741.28    17.97 o
 3   3   0      35.46       7.05    11.06 o
 4   3   0    4894.10    5052.51    61.43 o
 5   3   0       0.85      -5.16    14.37 o
 6   3   0    1683.30    1786.33    32.38 o
 7   3   0       2.15     -20.22    20.22 o
 8   3   0      14.13      57.63    43.53 o
 9   3   0    1153.58    1152.11    33.43 o
10   3   0      26.46      19.07    31.05 o
11   3   0     754.41     822.29    41.09 o
12   3   0    2451.15    2532.13    55.62 o
13   3   0     249.35     235.95    47.74 o
 0   4   0     356.42     509.90    34.66 o
 1   4   0   11624.85   12241.58   190.90 o
 2   4   0    6795.01    6877.47    95.67 o
 3   4   0   10070.62    9522.76   130.86 o
 4   4   0     283.35     313.52    14.14 o
 5   4   0    1762.78    1433.71    31.90 o
 6   4   0     985.01     927.18    34.23 o
 7   4   0    7060.07    6620.38   106.33 o
 8   4   0     338.90     401.19    26.46 o
 9   4   0   10697.62   10847.92   144.27 o
10   4   0     453.35     468.21    33.17 o
11   4   0    7083.52    7462.68   117.24 o
12   4   0      16.08     -41.03    41.03 o
13   4   0     957.74     924.27    50.89 o
 1   5   0    1130.26    1100.36    32.54 o
 2   5   0    6799.38    7057.50    98.94 o
 3   5   0    6001.10    5914.63    71.47 o
 4   5   0    2351.82    2323.56    32.36 o
 5   5   0     232.72     165.63    24.09 o
 6   5   0    1624.45    1689.58    28.80 o
 7   5   0    1402.61    1422.44    35.43 o
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 9   5   0     284.51     338.22    29.58 o
10   5   0    5281.96    5444.25    80.50 o
11   5   0    2759.41    2872.72    65.73 o
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 6   6   0    4022.89    3941.58     57.45 o
 7   6   0    3842.03    3926.62     58.53 o
 8   6   0    6937.90    7004.05     96.65 o
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10   6   0    5898.23    6261.15     90.51 o
11   6   0     532.11     590.49     40.17 o
12   6   0    3097.52    3301.07     67.01 o
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 3   7   0    3535.37    3763.71     43.82 o
 4   7   0     730.51     631.17     18.95 o
 5   7   0      74.41      67.25     20.32 o
 6   7   0     510.24     586.45     26.43 o
 7   7   0     515.48     517.28     28.67 o
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 9   7   0     277.76     278.52     33.31 o
10   7   0     440.62     392.58     36.21 o
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12   7   0     215.60     227.25     58.63 o
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 3   8   0     817.48     733.70     24.71 o
 4   8   0   10745.31   10593.96    119.74 o
 5   8   0       7.61      30.64     27.38 o
 6   8   0   15800.08   16584.67    215.64 o
 7   8   0     565.21     595.22     30.80 o
 8   8   0    4302.55    4323.73     72.94 o
 9   8   0      32.51      73.61     47.95 o
10   8   0    1644.57    1744.54     65.97 o
11   8   0     995.32    1092.58     55.27 o
12   8   0    1790.51    2047.36     94.45 o
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 2   9   0    1079.61    1159.21     27.66 o
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 5  10   0    6170.14    6196.50    120.04 o
 6  10   0    3945.56    3910.06     91.03 o
 7  10   0    2648.41    2738.82     69.77 o
 8  10   0     253.24     283.85     48.36 o
 9  10   0     136.91     163.46     52.23 o
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 -7   3  15     445.28     359.93     92.47 o
 -6   3  15     457.26     567.81     90.83 o
 -5   3  15      17.11       2.50     51.77 o
 -4   3  15    1106.59    1094.92     60.04 o
 -3   3  15     488.78     473.87     43.97 o
 -2   3  15    4925.88    4836.83    106.17 o
 -1   3  15     467.27     422.70     46.32 o
  0   3  15    6129.83    6317.86    113.09 o
  1   3  15      63.99      88.78     51.57 o
  2   3  15     964.76     986.58     48.96 o
  3   3  15     280.12     234.37     45.84 o
  4   3  15    1204.85    1194.29     63.82 o
```

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-8   4  15     5625.31     5476.79    310.59 o
-7   4  15       21.85      -23.36     95.40 o
-6   4  15     1047.08      905.94     72.56 o
-5   4  15       44.64      -26.97     56.34 o
-4   4  15      385.04      452.06     55.21 o
-3   4  15      610.55      551.42     44.79 o
-2   4  15     2272.63     2221.94     56.20 o
-1   4  15      222.89      275.50     46.13 o
 0   4  15     3539.37     3690.49     77.65 o
 1   4  15     1450.51     1402.71     49.72 o
 2   4  15     1667.30     1708.16     54.06 o
 3   4  15      519.26      439.45     47.12 o
 4   4  15      841.88     1007.04     85.58 o
 5   4  15       40.23       10.43     82.46 o
-7   5  15      584.80      169.40    114.02 o
-6   5  15     1324.74     1135.67     61.46 o
-5   5  15      177.39      179.01     57.69 o
-4   5  15     1108.89     1035.53     59.88 o
-3   5  15      253.05      295.44     59.67 o
-2   5  15     1813.53     1712.59     52.32 o
-1   5  15     1364.54     1405.67     53.98 o
 0   5  15      594.28      545.41     46.86 o
 1   5  15        9.91      -19.76     44.52 o
 2   5  15     1395.80     1342.76     61.11 o
 3   5  15        0.50      -11.72     44.13 o
 4   5  15     3886.70     4266.64    149.59 o
-7   6  15     3120.87     2892.61    117.78 o
-6   6  15      937.03     1032.05     70.84 o
-5   6  15      603.78      515.31     61.44 o
-4   6  15     2259.40     2240.98    114.21 o
-3   6  15      177.74       98.73     54.15 o
-2   6  15      604.71      613.53     46.45 o
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-6   7  15      346.29      269.09     69.24 o
-5   7  15      824.74      739.19     65.53 o
-4   7  15      499.96      471.28     59.23 o
-3   7  15      680.40      655.42     57.94 o
-2   7  15      173.11       62.34     54.57 o
-1   7  15     3915.14     3811.94     75.17 o
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 1   7  15     2652.09     2845.63     68.49 o
 2   7  15      503.14      421.96     46.70 o
 3   7  15     1597.61     1717.42    167.38 o
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-2   8  15      30.24      68.88     56.40 o
-1   8  15    2245.88    2362.36     81.30 o
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-1  10  15     330.87     385.38     62.06 o
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-7   1  16       0.40      37.77    114.02 o
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-5   1  16      98.22     199.57     58.98 o
-4   1  16     275.65     282.15     49.52 o
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-2   1  16      91.33      63.59     47.08 o
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 2   2  16    2161.38    2145.91     67.80 o
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 2   4  16     862.18     774.66    74.55 o
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 -4   1  17     1220.97     1154.84     63.00 o
 -3   1  17     1087.56     1086.22     59.19 o
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 -1   1  17     1907.67     1774.76     60.87 o
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 -4   2  17      428.09      379.17     67.53 o
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 -1   2  17       83.24       26.13     59.65 o
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 -4   3  17     2276.28     2213.23     95.65 o
 -3   3  17      757.99      640.93     57.88 o
 -2   3  17     2092.58     2172.73     67.28 o
 -1   3  17      776.83      821.72     90.54 o
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  2   3  17     3779.19     3909.53    183.34 o
 -4   4  17      414.78      292.73     88.56 o
 -3   4  17      218.29      118.21     73.62 o
 -2   4  17       66.02       85.53     54.51 o
 -1   4  17       98.88       62.44     55.93 o
  0   4  17        5.27        5.65     55.09 o
  1   4  17      185.02      170.91     54.75 o
 -3   5  17        4.02      -59.29     71.59 o
 -2   5  17     1357.91     1335.21     68.58 o
 -1   5  17       21.17       -4.57     57.14 o
  0   5  17      942.65     1028.01     60.14 o

===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_[Ru(tpm)(bpy)(H2O)](ClO4)2.MeOH, 3.11
_shelx_title ' 3.11 in P2(1)/c'
_shelx_refln_list_code          4
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_exptl_crystal_F_000      1456.00
_reflns_d_resolution_high  0.7998

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, y+1/2, -z+1/2'
 '-x, -y, -z'
 'x, -y-1/2, z-1/2'

_cell_length_a    14.9764
_cell_length_b    14.4923
_cell_length_c    14.3878
_cell_angle_alpha  90.000
_cell_angle_beta  116.902
_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
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   3   0   0     3488.09     3166.74     113.95 o
   4   0   0      247.42      144.85      27.54 o
   5   0   0    26044.88    25671.38     656.37 o
   6   0   0    10472.79    10218.07     268.14 o
   7   0   0     4903.26     5086.95     143.45 o
   8   0   0     2246.31     2192.53      86.97 o
   9   0   0      651.40      597.39      59.97 o
  10   0   0     3058.78     2935.18     105.77 o
  11   0   0    11176.95    10492.66     287.47 o
  12   0   0      401.71      384.84      77.03 o
  13   0   0     7509.19     7568.76     222.27 o
  14   0   0      484.59      483.81     127.95 o
  15   0   0     1759.43     1771.29     175.93 o
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   4   1   0     3091.06     3674.13     153.99 o
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  5  12   4      84.62     225.87     96.10 o
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  8  12   4     542.12     435.21    183.93 o
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 -5   1  15     827.86     681.56     64.25 o
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===END of fcf
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_reflns_d_resolution_high  0.8393

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 'x, -y-1/2, z-1/2'

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 4  21   1     236.26     168.63    309.48 o
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2	13	2	1716.37	1541.75	85.09	o
3	13	2	2023.74	2203.14	89.68	o
4	13	2	1895.97	1894.66	98.27	o
5	13	2	4315.05	4563.47	164.95	o
6	13	2	2329.31	2197.01	100.20	o
7	13	2	7477.34	7445.60	172.47	o
8	13	2	225.11	202.79	94.55	o
9	13	2	946.64	886.98	182.13	o
10	13	2	245.26	251.22	113.05	o
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3	14	2	6265.32	6192.78	145.11	o
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-7	15	2	634.01	578.49	94.15	o
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3	15	2	194.49	158.48	107.94	o
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5	15	2	252.97	177.78	87.85	o
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-1   7  13       2.41      98.76    225.27 o
 0   7  13    1361.29    1223.07    236.58 o
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-4   8  13    1064.05    1178.44    270.26 o
-3   8  13    1462.22     969.19    256.42 o
-2   8  13     724.78     916.59    323.37 o
-1   8  13      60.95    -263.55    632.85 o
 0   8  13      84.49     154.62    245.53 o
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 2   8  13    1578.23    1713.36    271.42 o
-5   9  13    1734.20    1618.35    266.65 o
-4   9  13     411.23     311.83    320.02 o
-3   9  13     566.43     305.29    261.07 o
-2   9  13     268.15     191.13    265.03 o
-1   9  13    2679.97    2669.00    296.05 o
 0   9  13    2171.31    1449.52    547.09 o
 1   9  13    3611.56    3498.41    291.44 o
-4  10  13     180.72     -89.88    257.37 o
-3  10  13     690.62     727.75    395.23 o
-2  10  13     266.21     643.70    261.84 o
-1  10  13     103.61     -63.73    353.07 o
-6   0  14       8.40     376.55    365.05 o
-5   0  14     681.96    1045.32    312.35 o
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 -3   0  14    3305.73    3427.24    404.33 o
 -2   0  14     235.98    -182.05    344.93 o
 -1   0  14    3224.49    3545.09    407.21 o
  0   0  14     221.53     301.81    370.80 o
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 -6   1  14    1297.50    1556.08    276.16 o
 -5   1  14       3.57      14.92    324.33 o
 -4   1  14       0.47     150.80    252.66 o
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  0   1  14     121.43     307.84    265.58 o
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  1   2  14    2437.48    2447.73    277.06 o
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 -5   3  14    1412.49    1868.02    338.22 o
 -4   3  14    3646.97    3729.25    426.85 o
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 -2   4  14    1129.39    1104.35    277.06 o
 -1   4  14    1307.48    1432.94    261.83 o
  0   4  14    1285.14    1439.20    256.43 o
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 -4   5  14    4124.55    4161.49    321.45 o
 -3   5  14     494.44     500.40    274.27 o
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 -1   5  14     259.17     262.94    276.90 o
  0   5  14    2461.77    2511.76    302.83 o
 -4   6  14     804.58     693.55    251.34 o
 -3   6  14       3.72    -238.87    238.87 o
 -2   6  14     662.90     572.36    285.79 o
 -1   6  14      80.70      53.09    262.88 o

===END of fcf
```

```
#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_[Ru(ttp)(bpy)(bpe)](PF6)2.MeOH, 3.14
_shelx_title ' 3.14 in P2(1)/c'
_shelx_refln_list_code          4
_shelx_F_calc_maximum      375.16
_exptl_crystal_F_000      2192.00
_reflns_d_resolution_high  0.7990

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, y+1/2, -z+1/2'
 '-x, -y, -z'
 'x, -y-1/2, z-1/2'

_cell_length_a    10.7421
_cell_length_b    21.6326
_cell_length_c    19.3655
_cell_angle_alpha  90.000
_cell_angle_beta   99.463
_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
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   2   0   0   12410.20   10373.15    322.65 o
   3   0   0   54893.50   59198.02   1832.02 o
   4   0   0    3647.00    4159.62     80.72 o
   5   0   0   18762.37   18910.57    348.71 o
   6   0   0     587.83     677.66     35.46 o
   7   0   0    5231.78    5141.10    103.37 o
   8   0   0    4529.37    4441.41    134.59 o
   9   0   0    2722.27    2939.82     72.43 o
  10   0   0     524.72     386.34     76.16 o
  11   0   0    4073.57    4014.39    162.02 o
  12   0   0    1037.85     929.17    110.78 o
  13   0   0       0.50      91.39    121.86 o
   1   1   0   54217.63   58921.77   1290.54 o
```

```
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 3   1   0   39886.34   45528.43    996.79 o
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 5   1   0    5215.67    5812.64     78.74 o
 6   1   0   10291.72   10628.58    140.90 o
 7   1   0    1805.27    1792.52     34.25 o
 8   1   0     819.55     743.57     27.99 o
 9   1   0    2124.91    2272.22     44.21 o
10   1   0     114.76      21.20     53.83 o
11   1   0     144.57     134.76     61.19 o
12   1   0     449.76     292.89     71.92 o
13   1   0     327.33     261.46     89.09 o
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 3   2   0    3388.06    4296.23     97.92 o
 4   2   0   14101.65   14304.14    186.49 o
 5   2   0   14774.71   15665.32    204.66 o
 6   2   0    3794.54    3881.70     55.93 o
 7   2   0    3601.01    3781.05     56.31 o
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 5   3   0    9919.39   11758.34    154.84 o
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 7   3   0     301.47     269.51     21.83 o
 8   3   0    5717.16    5753.25     81.93 o
 9   3   0     943.11     873.57     34.88 o
10   3   0      25.67      25.73     53.36 o
11   3   0    1820.84    1736.40     80.74 o
12   3   0     382.14     207.21     75.32 o
13   3   0     270.24     328.30     89.56 o
 0   4   0   12491.48   12866.53    296.16 o
 1   4   0     440.20     489.27     37.64 o
 2   4   0    1025.39     822.57     31.85 o
 3   4   0    1346.73    1466.00     32.38 o
 4   4   0    2677.01    2983.97     42.57 o
 5   4   0    1673.34    1796.96     30.04 o
 6   4   0      35.88      31.27     18.50 o
 7   4   0     154.15     193.98     21.93 o
 8   4   0    1740.74    1816.43     40.92 o
 9   4   0      48.81      27.76     33.16 o
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4	5	0	1002.77	1542.07	26.08	o
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6	5	0	3677.58	4005.17	57.83	o
7	5	0	691.17	714.98	25.21	o
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4	7	0	15763.04	16864.81	204.41	o
5	7	0	1759.14	1701.71	31.72	o
6	7	0	4896.23	5054.96	71.75	o
7	7	0	326.78	351.42	25.29	o
8	7	0	241.97	200.18	31.84	o
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6	8	0	1456.65	1537.99	31.42	o

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 3  17   0    1715.07    1840.70     40.75 o
 4  17   0    1899.99    2018.36     59.59 o
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 7  17   0      77.95      14.07     63.70 o
 8  17   0    1347.80    1316.06    108.01 o
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10  17   0     516.90     621.46    135.71 o
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 3  18   0    2924.03    2600.48    133.63 o
 4  18   0    2110.60    2103.91     68.52 o
 5  18   0    4250.87    4446.31    115.53 o
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 7  18   0    3864.37    3668.59    105.74 o
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 9  18   0     904.20    1119.48     83.71 o
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 4  19   0     642.32     510.00     48.93 o
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 7  19   0       0.22     -14.99     59.63 o
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 4  21   0       8.03      53.35     77.55 o
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 6  21   0     276.49     112.39     63.15 o
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```

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  4  22   0      89.26     116.12     57.12 o
  5  22   0    1290.68    1353.24     73.36 o
  6  22   0     153.66      80.58     68.05 o
  7  22   0     628.31     716.71     73.92 o
  1  23   0     994.59    1012.57     62.67 o
  2  23   0     125.88     144.77     57.27 o
  3  23   0      38.22      38.48     55.58 o
  4  23   0      57.29      23.51     59.63 o
  5  23   0       3.48     -18.80     66.04 o
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  0  24   0     111.80     661.91     90.01 o
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  4  24   0     150.40      49.51     66.04 o
  5  24   0     157.36     115.15     70.48 o
  6  24   0      19.47       0.00    112.16 o
  1  25   0     822.54     822.50     69.50 o
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  3  25   0      35.17     231.12     65.25 o
  4  25   0     530.32     640.35     73.88 o
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  0  26   0      42.79     243.72     96.93 o
  1  26   0      62.67     337.67     69.96 o
  2  26   0    1322.37    1423.47     78.82 o
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 3   6  21     1195.98     1113.68     48.36 o
 4   6  21      111.80       74.78     85.85 o
-7   7  21      483.08      509.81     54.60 o
-6   7  21      446.98      468.01    100.39 o
-5   7  21        1.69       11.25     40.72 o
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-3   7  21     1370.31     1335.25     74.66 o
-2   7  21        0.92      -38.70     38.70 o
-1   7  21      616.27      623.43     30.93 o
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-7   8  21     1208.12     1305.63     50.97 o
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-1   8  21      101.97       96.81     30.18 o
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 2   8  21      512.04      552.85     38.88 o
 3   8  21      176.97      222.29     40.93 o
-6   9  21        0.05       64.15     47.67 o
-5   9  21       36.88       79.18     44.12 o
-4   9  21       18.51        3.36     44.17 o
-3   9  21      774.12      803.39     32.87 o
-2   9  21       83.16       14.02     29.88 o
-1   9  21      484.17      400.34     33.17 o
 0   9  21        1.13      -10.86     33.02 o
 1   9  21      225.15      307.44     36.40 o
 2   9  21        1.46       16.30     38.95 o
 3   9  21        0.92        0.00     88.62 o
-6  10  21      197.20      248.99     32.69 o
-5  10  21      179.38      182.09     38.18 o
-4  10  21      176.88      218.37     31.12 o
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2	0	22	2327.97	2440.17	232.64	o
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  -2   7  23      124.81      161.80     36.58 o
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  -1   0  24     3039.58     3070.67     88.61 o
  -3   1  24      697.37      472.20     62.31 o
  -2   1  24       29.98       12.93     31.61 o
  -1   1  24       20.57      -41.88     44.27 o
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  -3   3  24      320.23      324.03     66.47 o
  -2   3  24      245.21      230.69     34.61 o

===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
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_shelx_title ' 1.71 in Pbca'
_shelx_refln_list_code          4
_shelx_F_calc_maximum      486.94
_exptl_crystal_F_000      2368.00
_reflns_d_resolution_high  0.8394

loop_
 _symmetry_equiv_pos_as_xyz
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 '-x+1/2, -y, z+1/2'
 '-x, y+1/2, -z+1/2'
 'x+1/2, -y+1/2, -z'
 '-x, -y, -z'
 'x-1/2, y, -z-1/2'
 'x, -y-1/2, z-1/2'
 '-x-1/2, y-1/2, z'

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_cell_length_b     8.7153
_cell_length_c    55.9700
_cell_angle_alpha  90.000
_cell_angle_beta   90.000
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_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
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 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
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 12   1   0      125.67       81.19     40.80 o
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  2   2   0    12468.34    12832.74    238.21 o
  4   2   0      369.31      321.09     32.53 o
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  8   5   0     2582.21     2696.43    129.39 o
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5	2	2	406.13	508.44	13.07	o
6	2	2	139.17	186.79	20.45	o
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13   1   6       1.70      74.34     31.76 o
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 9   2  17    4028.22    3864.16    84.68 o
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12   2  17       5.70      40.18    53.93 o
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 2   3  17     916.15    1098.26    66.73 o
 3   3  17      38.70      70.86    18.87 o
 4   3  17      26.97     103.73    18.24 o
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 6   3  17     409.96     574.06    22.55 o
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 8   3  17      57.59     174.55    29.94 o
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10   3  17     259.20     279.49    46.85 o
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12   3  17     197.79     186.84    52.22 o
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 1   4  17    3882.16    4288.49    64.16 o
 2   4  17    5214.56    5540.16    98.86 o
 3   4  17    1271.97    1368.06    36.65 o
 4   4  17     212.68     288.24    24.14 o
 5   4  17    1444.89    1506.29    40.51 o
 6   4  17     157.87     190.66    23.66 o
 7   4  17     860.63     852.71    26.81 o
 8   4  17     943.73     858.77    55.32 o
 9   4  17      61.38      83.37    46.71 o
10   4  17      38.05      90.81    48.44 o
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12   4  17       7.74      11.59    53.39 o
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 6   5  17      55.40      84.91    25.45 o
 7   5  17     861.47     844.87    46.66 o
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 4   6  17      75.16     109.73    32.46 o
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 7   6  17     135.82     172.11    39.52 o
 8   6  17      89.14     109.55    63.86 o
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 9   7  17      92.23     184.31     58.21 o
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 3   8  17       0.44      50.93     40.83 o
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 7   8  17       0.00     -76.06     76.06 o
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 3   9  17     136.85     206.08     85.62 o
 4   9  17       0.16     -47.75     47.75 o
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 4   0  18    7727.38    6961.73    184.87 o
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13   0  18     148.60     123.77     80.00 o
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 8   6  20       8.30     -51.59     51.59 o
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 5   1  21    1486.15    1715.46     36.12 o
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 7   1  21      77.48     106.24     23.37 o
 8   1  21    2122.42    1908.03     46.77 o
 9   1  21     602.12     636.84     31.22 o
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10   1  21      10.84      11.62     26.02 o
11   1  21      21.66     101.95     29.33 o
12   1  21      21.98      18.01     30.39 o
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 2   2  21     253.30     210.60     30.53 o
 3   2  21    1156.46    1222.42     57.98 o
 4   2  21     113.46     147.16     22.98 o
 5   2  21    1667.62    1732.26     41.38 o
 6   2  21     351.56     368.15     21.06 o
 7   2  21      21.81      33.62     21.76 o
 8   2  21    5530.20    5324.08     98.28 o
 9   2  21    1911.08    1909.94     49.60 o
10   2  21     136.95     144.19     27.70 o
11   2  21      13.63      47.97     29.13 o
12   2  21      79.95      66.12     36.61 o
 1   3  21    1524.47    1564.66     30.95 o
 2   3  21    2347.82    2350.40     39.85 o
 3   3  21    4789.85    4731.09     86.47 o
 4   3  21     952.79    1047.59     31.83 o
 5   3  21      90.98      86.05     22.15 o
 6   3  21      61.00      57.80     27.79 o
 7   3  21      24.49      30.14     21.79 o
 8   3  21    1980.06    1923.27     46.60 o
 9   3  21     187.66     168.24     26.47 o
10   3  21     125.53     123.55     33.00 o
11   3  21     535.68     454.16     53.01 o
12   3  21      84.78     103.32     53.81 o
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 1   4  21      11.23       3.80     21.04 o
 2   4  21     136.32     136.18     22.21 o
 3   4  21     310.71     367.59     25.80 o
 4   4  21     332.45     274.51     26.13 o
 5   4  21    2047.35    2049.27     49.04 o
 6   4  21     177.83     162.85     24.23 o
 7   4  21     213.69     226.17     21.78 o
 8   4  21      85.65      92.57     29.47 o
 9   4  21     212.04     219.69     50.30 o
10   4  21      89.67     100.09     48.48 o
11   4  21       0.39       8.41     51.83 o
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 2   5  21     329.88     234.98     40.70 o
 3   5  21     583.60     651.34     30.64 o
 4   5  21     723.97     802.89     36.97 o
 5   5  21      65.83     141.08     27.66 o
 6   5  21     511.56     446.52     31.18 o
 7   5  21      14.62      69.04     38.78 o
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 9   5  21       7.20      93.18     49.24 o
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 3   6  21     429.28     558.03     46.66 o
 4   6  21      54.45      86.80     38.58 o
 5   6  21     454.44     419.54     33.61 o
 6   6  21       8.23      27.56     37.71 o
 7   6  21      61.58     113.32     79.59 o
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10   6  21      21.69      -9.52     79.88 o
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 4   7  21       6.40       9.60     70.44 o
 5   7  21     266.87     271.45     55.85 o
 6   7  21      76.42      54.29     67.69 o
 7   7  21      95.47     113.61     74.14 o
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 4   8  21     260.79     305.90    134.30 o
 5   8  21     133.40     212.38     81.56 o
 6   8  21      75.79     144.22     83.71 o
 7   8  21     161.49     258.31     95.43 o
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 3   9  21      29.22      98.97     48.86 o
 4   9  21      94.87     156.57     49.70 o
 5   9  21      34.45      60.51     78.93 o
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 2   0  22    2595.79    2458.00     71.27 o
 3   0  22     619.05     773.10     30.35 o
 4   0  22    1934.86    1975.41     55.60 o
 5   0  22     242.74     226.57     24.34 o
 6   0  22     991.88    1079.99     46.12 o
 7   0  22    4497.58    4253.54    114.24 o
 8   0  22      64.83      92.16     34.36 o
 9   0  22    1673.44    1714.10     64.32 o
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 6   1  22      95.59     112.20     22.20 o
 7   1  22    2444.91    2292.69     51.31 o
 8   1  22     109.72     245.96     26.26 o
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 6   2  22     101.55      86.35     18.46 o
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12   2  22       8.91       2.12     36.23 o
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 3   3  22    2420.92    2530.84     52.98 o
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 6   3  22      18.65     -14.75     23.09 o
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 7   4  22     445.39     472.93     24.18 o
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 4   6  22     374.02     372.67     53.81 o
 5   6  22     272.31     345.08     30.86 o
 6   6  22      82.82     132.61     30.25 o
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 8   6  22      20.06      59.63     53.16 o
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 6   8  22      36.38      91.12     75.10 o
 7   8  22     211.81     197.08     86.82 o
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 2   9  22      70.47     106.66     49.30 o
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 4   9  22      59.33     101.73     50.66 o
 5   9  22     308.48     335.80     92.80 o
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 3   1  23     701.03     738.96     26.37 o
 4   1  23    2259.98    2352.85     43.99 o
 5   1  23      91.73     139.12     24.99 o
 6   1  23     271.73     344.39     24.84 o
 7   1  23       1.17      41.90     22.53 o
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 3   2  23      77.60     115.73    17.38 o
 4   2  23     416.82     489.10    24.05 o
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 6   2  23      91.84      73.52    18.94 o
 7   2  23       5.06      11.84    22.49 o
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 3   3  23     113.23     143.56    20.80 o
 4   3  23     330.25     419.64    39.77 o
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 6   3  23     247.16     251.79    25.95 o
 7   3  23    1708.70    1765.49    43.92 o
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 6   5  23      12.25       4.28    27.02 o
 7   5  23     294.25     344.73    39.81 o
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 4   6  23      192.15      248.28     51.30 o
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 8   6  23      189.75      251.95     69.12 o
 9   6  23       78.90      128.92     57.72 o
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 5   7  23       69.62       72.83     46.92 o
 6   7  23       55.18      119.59     71.03 o
 7   7  23      232.31      340.34     81.08 o
 8   7  23       60.57       35.64     77.97 o
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 3   8  23      772.05      784.32     56.19 o
 4   8  23       69.21       21.53     45.03 o
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 7   2  24     595.57     620.57     29.04 o
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 6   6  24     233.59     222.50     31.08 o
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 8   7  24     193.96     289.88     82.28 o
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8	3	29	0.46	61.31	26.77	o
9	3	29	126.25	122.25	28.22	o
10	3	29	3.02	-1.48	30.50	o

```
11   3  29      18.27      33.91     36.99 o
 0   4  29    3797.46    3481.63    140.15 o
 1   4  29    1746.88    1672.98     44.79 o
 2   4  29      68.63     121.36     35.84 o
 3   4  29      92.30      79.42     37.46 o
 4   4  29     588.39     592.85     30.81 o
 5   4  29     143.06     168.81     26.60 o
 6   4  29     159.43     179.79     27.01 o
 7   4  29      41.84       5.37     29.83 o
 8   4  29       0.47      40.48     25.71 o
 9   4  29     896.53     884.95     45.17 o
10   4  29       3.10      -0.47     71.15 o
11   4  29       0.01      31.04     54.88 o
 1   5  29     237.41     252.19     40.11 o
 2   5  29     218.98     242.06     41.68 o
 3   5  29      45.49      35.65     28.91 o
 4   5  29      28.44      57.17     28.60 o
 5   5  29    1392.77    1385.00     44.83 o
 6   5  29    2849.39    2551.99     62.77 o
 7   5  29       4.30     -26.42     30.95 o
 8   5  29      89.82     100.71     34.54 o
 9   5  29      35.20     -35.70     52.54 o
10   5  29       0.34     -35.49     53.25 o
 0   6  29   10186.36    9682.88    339.62 o
 1   6  29    1916.49    1925.53     74.88 o
 2   6  29    4075.81    3940.23    129.75 o
 3   6  29    1666.02    1604.92     68.33 o
 4   6  29     564.63     571.76     48.55 o
 5   6  29     230.65     181.21     42.25 o
 6   6  29       6.47      19.52     31.64 o
 7   6  29      13.46       4.86     41.09 o
 8   6  29      19.05     -37.59     58.36 o
 9   6  29     115.10     112.51     73.07 o
 1   7  29      38.16      39.28     48.19 o
 2   7  29     114.74     111.64     51.36 o
 3   7  29     150.65     131.14     47.20 o
 4   7  29     141.56     161.63     45.96 o
 5   7  29     161.49     162.08     44.65 o
 6   7  29      33.49     -21.29     69.60 o
 7   7  29     376.12     452.04     86.34 o
 0   8  29      27.02      -1.91     73.90 o
 1   8  29      13.88      21.08     76.77 o
 2   8  29     385.44     376.54     58.60 o
 3   8  29     525.42     529.25     57.68 o
 4   8  29       2.61       9.66     47.86 o
 5   8  29      38.70     -53.53     53.53 o
 6   8  29       0.02     115.52     89.69 o
 1   9  29      37.96     166.49     50.73 o
 2   9  29     200.46     235.95     52.77 o
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 3   9  29     219.82     165.27    143.14 o
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 1   0  30     233.03     386.84    133.70 o
 2   0  30   10448.82   11038.52    261.12 o
 3   0  30    6833.30    7325.72    221.71 o
 4   0  30      53.33      52.70     23.12 o
 5   0  30     771.23     835.78     43.11 o
 6   0  30     599.79     538.61     39.74 o
 7   0  30    2655.77    2287.55     84.55 o
 8   0  30    2045.87    1959.34     73.82 o
 9   0  30    6430.73    5769.77    155.41 o
10   0  30      28.87      47.63     40.80 o
11   0  30       6.88      22.31     44.91 o
12   0  30      73.03      89.72     74.86 o
 1   1  30     637.00     798.09     27.75 o
 2   1  30     119.68     188.64     36.49 o
 3   1  30    3016.36    3056.75     54.97 o
 4   1  30     594.21     592.19     27.67 o
 5   1  30    1457.53    1374.04     34.64 o
 6   1  30      29.56      34.80     29.27 o
 7   1  30    2011.92    1908.87     48.60 o
 8   1  30    2069.31    2097.58     53.23 o
 9   1  30     245.73     335.32     31.14 o
10   1  30     453.16     471.55     34.30 o
11   1  30      86.00      52.83     31.12 o
12   1  30     166.78     163.27     34.87 o
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 2   2  30     281.28     277.12     20.23 o
 3   2  30     411.12     341.57     24.79 o
 4   2  30    3236.76    3416.46     69.18 o
 5   2  30     680.06     607.90     30.19 o
 6   2  30      23.60      86.65     24.10 o
 7   2  30    1293.34    1249.36     38.77 o
 8   2  30     176.27     293.96     28.01 o
 9   2  30       4.32     122.35     28.82 o
10   2  30       0.93      18.86     30.36 o
11   2  30      10.75      27.56     30.80 o
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 2   3  30     513.53     462.40     27.57 o
 3   3  30     219.59     273.05     36.98 o
 4   3  30    1968.73    1888.45     47.49 o
 5   3  30      37.69      88.08     26.14 o
 6   3  30      74.37      52.15     27.16 o
 7   3  30     899.01     895.08     35.07 o
 8   3  30     686.89     546.03     32.23 o
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10   3  30      39.86       7.99     30.45 o
11   3  30      10.20       4.01     37.11 o
```

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 3   4  30      189.59      240.77     32.92 o
 4   4  30     1404.38     1400.78     42.72 o
 5   4  30        0.45        2.04     25.36 o
 6   4  30      859.94      752.82     35.41 o
 7   4  30       16.14       24.62     31.51 o
 8   4  30        1.03        7.61     26.50 o
 9   4  30       77.26      115.37     30.39 o
10   4  30       59.39       38.85     52.67 o
 1   5  30      195.71      233.40     38.55 o
 2   5  30     1260.37     1018.71     54.71 o
 3   5  30      233.12      256.84     28.84 o
 4   5  30       71.89       49.25     28.03 o
 5   5  30      559.89      566.13     34.86 o
 6   5  30      437.14      418.54     34.21 o
 7   5  30       99.18      130.23     44.45 o
 8   5  30      122.91       97.58     42.62 o
 9   5  30       36.70        5.30     55.00 o
10   5  30       10.28       13.47     55.54 o
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 2   6  30     1437.42     1447.48     66.29 o
 3   6  30      552.89      564.97     87.30 o
 4   6  30        0.16       78.85     42.25 o
 5   6  30      246.56      261.36     46.23 o
 6   6  30        5.87      -33.43     33.43 o
 7   6  30       38.18       50.63     48.78 o
 8   6  30       11.57       43.67     55.67 o
 9   6  30        0.92       86.73     63.30 o
 1   7  30      150.62      114.91     48.85 o
 2   7  30      103.64      178.55     47.12 o
 3   7  30      669.33      527.72     52.91 o
 4   7  30      442.78      493.07     53.61 o
 5   7  30      217.02      183.64     56.56 o
 6   7  30       72.33      114.56     80.60 o
 7   7  30      502.61      617.06     92.56 o
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 2   8  30        5.04       15.23     53.77 o
 3   8  30      350.91      355.98     55.60 o
 4   8  30       68.04      150.75     69.60 o
 5   8  30       13.55       -0.90     51.63 o
 6   8  30      112.29       57.88     89.45 o
 1   9  30        0.02       61.83     50.81 o
 2   9  30       33.32       80.20     52.74 o
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 2   1  31      124.76      223.83     28.31 o
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 5   1  31     983.07    1064.54    30.52 o
 6   1  31     427.75     561.37    29.44 o
 7   1  31       8.17      54.26    26.71 o
 8   1  31     699.34     960.40    41.32 o
 9   1  31     834.63     768.49    37.12 o
10   1  31     576.28     632.93    43.15 o
11   1  31      31.96       4.41    31.51 o
 1   2  31    2474.85    2431.62    42.46 o
 2   2  31     774.09     838.38    27.56 o
 3   2  31     124.99     155.23    23.19 o
 4   2  31       5.85      68.04    25.88 o
 5   2  31      12.37       8.98    23.05 o
 6   2  31     750.57     785.18    37.55 o
 7   2  31    1457.63    1485.90    43.19 o
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 9   2  31    1485.30    1413.91    44.82 o
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 2   3  31      49.52      16.27    22.85 o
 3   3  31      37.04      77.32    23.46 o
 4   3  31     425.68     471.08    28.45 o
 5   3  31     988.21     934.79    35.08 o
 6   3  31       5.76     -23.27    25.10 o
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10   3  31     364.03     372.70    34.36 o
11   3  31       4.26      29.63    37.89 o
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 3   4  31     228.18     243.12    27.52 o
 4   4  31     382.55     384.67    44.33 o
 5   4  31     234.97     220.80    31.57 o
 6   4  31     202.78     290.43    30.52 o
 7   4  31     254.00     234.42    31.93 o
 8   4  31       4.36     -26.83    26.83 o
 9   4  31      54.71      71.14    30.21 o
10   4  31      49.39      50.58    53.51 o
 1   5  31    4566.59    3937.44   182.01 o
 2   5  31     209.69     185.45    42.06 o
 3   5  31     403.36     402.85    49.46 o
 4   5  31      71.54      43.85    29.08 o
 5   5  31     529.58     607.29    35.60 o
 6   5  31     173.45     138.34    31.46 o
 7   5  31     125.75     105.91    35.62 o
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 9   5  31      69.64      41.77     55.55 o
10   5  31      16.44      -2.05     58.73 o
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 1   6  31     484.79     479.78     49.10 o
 2   6  31      14.54      94.59     43.55 o
 3   6  31     132.97     197.26     44.24 o
 4   6  31      42.88     113.89     76.77 o
 5   6  31     672.62     575.12     84.67 o
 6   6  31       3.59      12.42     36.28 o
 7   6  31     477.66     584.73     72.65 o
 8   6  31      54.93      15.83     58.90 o
 9   6  31       0.04      55.36     62.01 o
 1   7  31     132.71      76.88     49.64 o
 2   7  31      24.47     104.52     50.65 o
 3   7  31     354.45     386.16     54.16 o
 4   7  31     113.64     158.05     49.02 o
 5   7  31       0.32      58.16     46.55 o
 6   7  31     353.02     335.75     52.04 o
 7   7  31      46.80     153.31     87.06 o
 0   8  31     643.15     504.65     88.02 o
 1   8  31     486.80     550.57     61.07 o
 2   8  31      58.81      -4.59     51.71 o
 3   8  31      40.73      76.01     55.22 o
 4   8  31     601.94     510.18     59.92 o
 5   8  31     206.89     202.88     54.86 o
 1   9  31       2.25      19.82     52.57 o
 2   9  31       8.12     -43.53     52.93 o
 0   0  32     946.93     982.76     60.99 o
 1   0  32    3656.86    3375.22     93.69 o
 2   0  32     167.36     207.89     29.16 o
 3   0  32    2778.83    3380.52     95.72 o
 4   0  32    2005.79    2299.68     59.92 o
 5   0  32     211.52     263.68     33.57 o
 6   0  32     958.40     908.19     48.11 o
 7   0  32    1026.07     892.63     51.55 o
 8   0  32     536.79     555.43     47.60 o
 9   0  32     904.92    1012.92     56.92 o
10   0  32       8.61     -40.26     40.26 o
11   0  32     145.70     143.60     47.41 o
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 2   1  32     372.69     593.28     23.93 o
 3   1  32    1305.78    1526.95     40.98 o
 4   1  32       1.73      16.05     23.17 o
 5   1  32     325.63     269.59     24.79 o
 6   1  32     314.25     408.71     28.53 o
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 9   1  32    1305.52    1170.64     42.99 o
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 3   2  32        1.33       51.72     24.40 o
 4   2  32      617.89      711.33     30.81 o
 5   2  32       55.26       84.49     26.30 o
 6   2  32        7.77       58.99     24.35 o
 7   2  32      431.59      582.19     32.62 o
 8   2  32      101.62      138.99     28.57 o
 9   2  32      109.34      203.18     32.66 o
10   2  32      203.81      234.07     31.30 o
11   2  32       83.74       91.35     31.71 o
 1   3  32      861.59      935.75     30.48 o
 2   3  32      142.08      130.42     24.32 o
 3   3  32      584.04      598.90     29.61 o
 4   3  32     1003.19      969.68     35.76 o
 5   3  32      349.14      345.85     29.36 o
 6   3  32        9.26       42.98     26.86 o
 7   3  32      152.56      121.41     26.12 o
 8   3  32       70.84       85.05     26.82 o
 9   3  32        0.45       23.85     27.94 o
10   3  32        7.08       -2.79     30.85 o
11   3  32        0.42      -25.83     38.49 o
 0   4  32      588.01      579.51     65.77 o
 1   4  32      324.02      299.41     28.70 o
 2   4  32       84.93      115.75     27.46 o
 3   4  32       67.31       78.03     33.28 o
 4   4  32      578.97      675.96     33.56 o
 5   4  32     1460.50     1252.54     41.57 o
 6   4  32      123.91      172.25     28.24 o
 7   4  32      217.27      120.57     41.67 o
 8   4  32        5.80       25.34     27.64 o
 9   4  32       38.65       24.92     29.76 o
10   4  32       53.21       32.27     53.75 o
 1   5  32        0.00      376.70     60.99 o
 2   5  32       56.12      153.26     41.94 o
 3   5  32       31.37       24.90     33.40 o
 4   5  32      232.18      186.03     29.94 o
 5   5  32      102.84       75.73     30.09 o
 6   5  32       12.50       38.42     31.19 o
 7   5  32       36.49        3.52     35.73 o
 8   5  32        8.55       24.20     34.74 o
 9   5  32        3.84      -21.87     51.10 o
 0   6  32      293.34      365.46     67.45 o
 1   6  32      372.94      299.53     44.56 o
 2   6  32        0.24      -17.51     42.30 o
 3   6  32      264.48      230.81     52.38 o
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 4   6  32     381.77     490.35     51.74 o
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 7   6  32     669.69     655.09     52.15 o
 8   6  32      90.18      45.60     60.46 o
 1   7  32     374.13     335.27     56.80 o
 2   7  32     433.49     392.91     53.04 o
 3   7  32     425.54     490.03     76.06 o
 4   7  32     668.25     714.78    100.09 o
 5   7  32     702.50     722.29     56.06 o
 6   7  32      13.89      -7.44     83.35 o
 7   7  32     115.25     139.20     87.30 o
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 3   8  32      20.19      14.07     76.77 o
 4   8  32     139.79     251.98     59.57 o
 5   8  32     146.23      88.04     56.87 o
 1   9  32     195.24     245.47     55.98 o
 1   1  33       1.35       8.13     26.24 o
 2   1  33    3506.69    3716.48     64.95 o
 3   1  33     983.16    1032.63     29.40 o
 4   1  33     690.77     837.10     31.28 o
 5   1  33     163.21     160.95     24.34 o
 6   1  33    1349.41    1427.52     41.86 o
 7   1  33    1405.80    1313.03     41.75 o
 8   1  33     581.81     756.62     35.80 o
 9   1  33    1304.15    1346.22     45.29 o
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 4   2  33     207.67     241.62     26.37 o
 5   2  33     240.86     240.87     29.09 o
 6   2  33    1346.70    1373.87     41.58 o
 7   2  33    3403.11    3414.43     77.22 o
 8   2  33      41.45      64.04     28.06 o
 9   2  33    1109.10    1084.81     41.44 o
10   2  33    1044.85    1037.95     41.92 o
11   2  33      64.74      70.58     31.79 o
 1   3  33      81.92     123.53     22.40 o
 2   3  33      32.68      47.28     23.15 o
 3   3  33      82.53     128.38     32.42 o
 4   3  33       0.59      11.84     24.19 o
 5   3  33       1.35      64.68     27.06 o
 6   3  33     427.13     426.14     32.83 o
 7   3  33     731.97     710.21     37.59 o
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 8   3  33     621.43     652.62     41.22 o
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 2   4  33     309.83     300.22     29.00 o
 3   4  33       5.37      -1.52     26.95 o
 4   4  33      98.71      32.96     26.02 o
 5   4  33     634.05     760.54     35.91 o
 6   4  33     684.64     653.37     34.89 o
 7   4  33      17.53     -22.69     34.90 o
 8   4  33     110.81      88.64     27.51 o
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10   4  33     254.14     217.58     57.50 o
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 5   5  33      75.96      53.96     39.46 o
 6   5  33     194.56     208.25     33.87 o
 7   5  33     140.98     141.90     44.12 o
 8   5  33     300.26     244.60     32.20 o
 9   5  33      75.08     137.65     54.74 o
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 1   6  33    1588.53    1494.24     67.86 o
 2   6  33     669.98     618.21    116.36 o
 3   6  33      46.92      85.71     43.52 o
 4   6  33     216.26     304.54     47.87 o
 5   6  33    1073.97     892.87     59.19 o
 6   6  33     172.80     140.31     40.56 o
 7   6  33       8.26      34.16     35.92 o
 8   6  33       4.03     -28.72     59.93 o
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 3   7  33      26.55      68.50     48.83 o
 4   7  33     679.62     521.09     56.08 o
 5   7  33     434.73     466.73     54.46 o
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 1   8  33       0.84       8.27     54.67 o
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 3   8  33      30.87       7.91     56.09 o
 4   8  33     225.19     201.58     75.58 o
 5   8  33       0.65      49.63     57.28 o
 0   0  34     927.51     988.26     67.45 o
 1   0  34    1633.14    1750.93     77.61 o
 2   0  34       6.25       9.02     28.99 o
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 3   0  34    5780.26    5997.89   153.22 o
 4   0  34       3.17      -2.18    25.24 o
 5   0  34    1308.81    1233.70    53.01 o
 6   0  34    3198.24    3107.56    93.93 o
 7   0  34    1584.47    1544.48    64.90 o
 8   0  34    2690.16    2501.83    85.83 o
 9   0  34       7.35      26.17    55.97 o
10   0  34      59.63       8.36    42.24 o
11   0  34     461.79     377.43    51.89 o
 1   1  34      70.24      69.08    20.70 o
 2   1  34    1241.19    1390.84    34.34 o
 3   1  34    1125.54    1163.74    32.22 o
 4   1  34     774.83     772.03    31.12 o
 5   1  34     928.41     816.46    40.98 o
 6   1  34      48.41      72.92    25.75 o
 7   1  34     972.57    1119.65    40.24 o
 8   1  34     930.69     914.70    54.62 o
 9   1  34    1153.88    1204.74    43.71 o
10   1  34       0.93     -18.87    30.65 o
11   1  34      10.19      -5.14    32.70 o
 0   2  34    2760.37    2762.53    67.89 o
 1   2  34      63.86      93.39    17.17 o
 2   2  34      88.62     157.25    21.89 o
 3   2  34      55.63     142.46    23.94 o
 4   2  34     452.60     470.08    29.07 o
 5   2  34     444.54     461.22    29.77 o
 6   2  34    2424.14    2506.48    58.10 o
 7   2  34    1188.66    1464.94    48.05 o
 8   2  34     706.67     700.91    35.52 o
 9   2  34      81.28     134.98    30.57 o
10   2  34       7.61      31.54    30.05 o
11   2  34     178.94     204.69    39.29 o
 1   3  34     552.18     608.57    29.63 o
 2   3  34    1920.85    1863.91    48.03 o
 3   3  34    1657.40    1615.11    44.73 o
 4   3  34      25.77      73.29    26.11 o
 5   3  34    2139.17    1846.57    50.10 o
 6   3  34      48.77      55.07    31.24 o
 7   3  34       0.01      38.39    27.72 o
 8   3  34       1.90      36.91    28.43 o
 9   3  34      28.14      24.27    30.61 o
10   3  34      61.20     -20.22    31.81 o
11   3  34      71.47     144.58    41.31 o
 0   4  34    3332.74    2982.00   129.63 o
 1   4  34     225.97     218.14    35.06 o
 2   4  34       2.49      40.82    28.48 o
 3   4  34     127.32     109.04    27.72 o
 4   4  34       5.45      18.81    26.80 o
 5   4  34       1.12     109.81    27.04 o
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 6   4  34       0.43      24.95     29.18 o
 7   4  34     328.73     291.70     37.01 o
 8   4  34      15.50     -24.75     28.55 o
 9   4  34     154.05     105.10     31.97 o
10   4  34     277.27     273.45     41.92 o
 1   5  34     764.76     884.91     52.22 o
 2   5  34     739.60     642.02     70.91 o
 3   5  34      99.89      82.58     40.81 o
 4   5  34    1513.17    1351.71     47.44 o
 5   5  34     574.48     503.11     36.03 o
 6   5  34     983.34     886.63     42.51 o
 7   5  34      25.22      65.01     36.44 o
 8   5  34      21.19      42.42     30.78 o
 9   5  34       0.44     -45.78     53.31 o
 0   6  34     393.69     477.15     70.79 o
 1   6  34    1824.53    1837.33     75.25 o
 2   6  34    2505.79    2336.04     85.24 o
 3   6  34     144.32     179.04     43.03 o
 4   6  34     104.46     215.40     45.42 o
 5   6  34      52.91      43.90     48.06 o
 6   6  34      44.48      67.03     40.49 o
 7   6  34     221.28     281.46     38.82 o
 8   6  34      33.66      18.09     60.46 o
 1   7  34     436.75     455.01     55.84 o
 2   7  34     266.72     331.53     50.59 o
 3   7  34     117.59      79.49     48.42 o
 4   7  34       5.84      21.71     50.70 o
 5   7  34      79.10     145.42     78.45 o
 6   7  34      44.36      86.54     50.05 o
 7   7  34      46.34      93.99     91.60 o
 0   8  34     569.91     453.95     88.97 o
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 2   8  34       5.21     -36.77     57.18 o
 3   8  34       3.42      17.68     53.94 o
 4   8  34      95.88      45.18     53.28 o
 5   8  34     332.80     301.10     59.29 o
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 2   1  35     210.78     332.76     32.62 o
 3   1  35     357.61     376.85     27.39 o
 4   1  35      22.70      61.35     23.57 o
 5   1  35       5.14      32.17     23.91 o
 6   1  35       0.78     -25.14     25.14 o
 7   1  35    2748.26    2688.42     63.05 o
 8   1  35    1212.69    1383.70     45.62 o
 9   1  35     204.10     267.10     36.73 o
10   1  35     261.37     335.81     34.70 o
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 1   2  35     600.12     728.60     27.48 o
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 6   2  35    2810.71    2766.67     62.90 o
 7   2  35    3261.14    3328.94     71.52 o
 8   2  35     110.66     150.58     28.81 o
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10   2  35     259.59     260.46     34.54 o
11   2  35      34.38      26.27     32.70 o
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 3   3  35     109.11     134.32     25.65 o
 4   3  35      55.65      38.80     27.12 o
 5   3  35      81.30     108.92     27.55 o
 6   3  35       1.78       1.27     31.04 o
 7   3  35      12.48       7.67     27.51 o
 8   3  35      51.40      92.67     29.46 o
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10   3  35      71.37      58.23     33.53 o
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 2   4  35    1370.34    1300.50     42.18 o
 3   4  35      26.35      24.78     28.10 o
 4   4  35     387.97     295.70     29.54 o
 5   4  35    2858.51    2562.06     61.91 o
 6   4  35     158.24     238.74     31.79 o
 7   4  35      30.59      73.80     34.34 o
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 9   4  35      29.39      42.89     32.22 o
10   4  35      83.56     103.61     40.59 o
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 3   5  35       9.62      20.79     62.54 o
 4   5  35     516.63     414.77     35.06 o
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 6   5  35     881.85     826.07     41.62 o
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 9   5  35      61.69      59.55     54.86 o
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 2   6  35      57.11     146.22     66.49 o
 3   6  35     255.86     262.77     45.61 o
 4   6  35       0.41      35.10     48.44 o
 5   6  35     117.57     163.46     51.93 o
 6   6  35      32.48      69.51     42.63 o
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 8   6  35      61.50      66.88     61.64 o
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 4   7  35     231.00     245.18     51.39 o
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 6   7  35     201.72     345.43     55.06 o
 7   7  35       8.25     122.70     93.52 o
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 2   8  35       0.61     -35.57     52.44 o
 3   8  35     338.58     340.68     58.73 o
 4   8  35      69.88     142.69     55.34 o
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 4   0  36     244.00     219.14     28.35 o
 5   0  36     294.99     277.31     46.76 o
 6   0  36     250.13     192.82     39.47 o
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 8   0  36     222.34     202.61     76.06 o
 9   0  36     468.05     427.37     68.52 o
10   0  36       0.12      23.44     43.84 o
11   0  36       5.68      24.51     80.24 o
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 4   1  36     117.26     125.92     24.88 o
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 7   1  36      19.94     115.82     28.33 o
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 9   1  36       1.23      18.81     31.07 o
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 3   2  36    1445.02    1464.11     42.51 o
 4   2  36     778.45     800.75     34.09 o
 5   2  36      41.24     126.37     27.78 o
 6   2  36    1337.08    1404.45     43.12 o
 7   2  36      56.03     111.57     33.97 o
 8   2  36       9.18       3.17     27.44 o
 9   2  36       3.46     -11.17     30.10 o
10   2  36     189.20     209.53     34.28 o
11   2  36     121.17     171.24     34.97 o
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 3   3  36      80.58     106.65     25.93 o
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 8   3  36     883.76    867.04    66.38 o
 9   3  36      77.34    132.18    32.08 o
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 2   4  36     223.34    245.29    28.54 o
 3   4  36       3.97     14.25    26.73 o
 4   4  36      50.12     75.78    27.10 o
 5   4  36     712.51    607.97    34.32 o
 6   4  36     517.08    502.36    35.20 o
 7   4  36     581.13    466.73    57.98 o
 8   4  36     245.74    219.37    38.20 o
 9   4  36     185.26    214.64    33.60 o
10   4  36     334.84    443.68    45.24 o
 1   5  36     422.80    519.23    60.27 o
 2   5  36      37.50     78.54    54.41 o
 3   5  36     138.53    197.85    90.53 o
 4   5  36     164.22    190.90    32.47 o
 5   5  36     199.02    334.48    34.62 o
 6   5  36       2.03     54.65    33.64 o
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 8   5  36     309.92    317.42    34.51 o
 9   5  36      57.39     56.06   119.11 o
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 2   6  36     203.72    232.00    46.64 o
 3   6  36     249.22    326.31    49.44 o
 4   6  36     391.65    310.21    53.76 o
 5   6  36      54.64     50.14    51.53 o
 6   6  36      23.60    -23.28    54.24 o
 7   6  36       1.15     28.58    42.20 o
 8   6  36      52.51      4.24    61.31 o
 1   7  36      50.64     28.27    47.18 o
 2   7  36       7.10     -0.95    51.93 o
 3   7  36      18.51     34.14    54.17 o
 4   7  36     245.04    306.46    56.53 o
 5   7  36     214.94    241.80    54.21 o
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 0   8  36    1331.53   1161.18   106.19 o
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 2   8  36     534.92    447.04    63.64 o
 3   8  36     241.36    218.22    57.58 o
 4   8  36     178.20    104.16    56.34 o
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 2   1  37       0.14     67.22    21.80 o
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 3   1  37     199.25     232.08     22.40 o
 4   1  37     379.40     445.87     28.98 o
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 6   1  37     232.45     264.99     28.30 o
 7   1  37     453.90     498.84     33.30 o
 8   1  37    1416.98    1494.61     54.50 o
 9   1  37      73.10      36.84     31.31 o
10   1  37      33.16      50.26     38.35 o
11   1  37      10.85      27.63     33.77 o
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 2   2  37     753.94     948.04     48.05 o
 3   2  37     124.05     232.38     26.79 o
 4   2  37      39.38     122.03     35.87 o
 5   2  37     969.68    1109.94     39.30 o
 6   2  37       9.68      99.99     26.38 o
 7   2  37      11.75      13.04     26.84 o
 8   2  37     394.41     423.66     33.63 o
 9   2  37     152.03     176.14     32.26 o
10   2  37      74.36      -0.58     33.16 o
11   2  37     532.56     514.03     39.35 o
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 2   3  37     389.00     405.14     29.92 o
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 4   3  37      26.26      77.87     27.86 o
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 6   3  37       0.17      27.40     39.09 o
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10   3  37     116.17     125.50     35.40 o
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 2   4  37     137.49     191.64     29.29 o
 3   4  37     638.40     666.43     34.60 o
 4   4  37     117.60      61.98     27.37 o
 5   4  37     287.91     333.08     33.95 o
 6   4  37    1634.84    1535.39     49.58 o
 7   4  37     228.92     248.56     37.09 o
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10   4  37     151.16     276.77     44.11 o
 1   5  37    1466.37    1358.19     90.05 o
 2   5  37      86.94      89.90     63.86 o
 3   5  37      48.45       1.51     43.41 o
 4   5  37       7.06      30.87     37.52 o
 5   5  37    1912.93    1855.81     55.26 o
 6   5  37       0.14      12.63     33.43 o
 7   5  37    1092.87    1167.69     53.87 o
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 8   5  37      14.29      76.32     34.05 o
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 2   6  37     198.79     146.56     45.82 o
 3   6  37     343.01     309.58     49.59 o
 4   6  37      36.26     -12.57     50.39 o
 5   6  37      66.84     126.20     64.58 o
 6   6  37      95.88     139.01     54.53 o
 7   6  37     380.85     409.96     47.20 o
 8   6  37       0.00     -41.03     60.97 o
 1   7  37     312.90     293.94     51.56 o
 2   7  37       1.07      29.65     52.78 o
 3   7  37     493.78     412.44    117.79 o
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 6   7  37      95.63      87.50     54.30 o
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 2   8  37     124.45     194.74    111.93 o
 3   8  37     152.42     157.01     61.09 o
 4   8  37      79.21      87.80     56.51 o
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 2   0  38      44.12     -19.16     46.04 o
 3   0  38     122.10     124.82     35.58 o
 4   0  38    1356.33    1379.20     64.58 o
 5   0  38     104.36      99.52     44.37 o
 6   0  38     704.99     766.83     52.26 o
 7   0  38     317.08     365.44     79.17 o
 8   0  38    4178.31    4020.88    120.30 o
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 3   1  38      87.20     138.97     26.82 o
 4   1  38    1019.30     913.87     34.53 o
 5   1  38     826.01     817.69     33.59 o
 6   1  38       2.74      49.77     26.81 o
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 8   1  38      52.57     136.56     30.39 o
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10   1  38      63.14      60.60     32.67 o
11   1  38       4.09      -3.75     33.95 o
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 3   2  38    1939.03    2111.45     76.88 o
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 4   2  38     786.63     854.19     35.35 o
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 9   2  38     753.26     859.92     40.53 o
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 3   3  38     108.97     142.27     27.52 o
 4   3  38     383.26     332.91     30.96 o
 5   3  38       3.92      75.36     30.59 o
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 7   3  38      45.27      39.72     28.88 o
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 4   4  38      16.24      33.68     27.61 o
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 6   4  38     164.81     239.25     33.59 o
 7   4  38     603.43     515.44     41.40 o
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 2   6  38      23.13       7.66     44.37 o
 3   6  38      51.33      65.03     56.80 o
 4   6  38      15.45      20.85     48.98 o
 5   6  38     448.18     470.16     59.20 o
 6   6  38      74.07      77.88     55.48 o
 7   6  38       0.43     102.25     56.13 o
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 4   7  38       0.10     -32.38     54.19 o
 5   7  38      74.87      39.80     54.60 o
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 3   8  38     394.40     289.96     64.24 o
 1   1  39     551.95     613.84     29.97 o
 2   1  39    1133.27    1078.41     32.52 o
 3   1  39     570.48     638.67     30.66 o
 4   1  39       0.00      57.45     26.94 o
 5   1  39      48.99     168.42     34.63 o
 6   1  39     238.30     259.72     29.22 o
 7   1  39     132.35     174.85     30.44 o
 8   1  39      27.05      73.33     30.06 o
 9   1  39     129.86     138.92     32.18 o
10   1  39       0.50      -0.80     32.17 o
 0   2  39    2120.40    2258.10     61.76 o
 1   2  39       2.42      63.07     23.75 o
 2   2  39     821.01     930.81     35.01 o
 3   2  39    1242.22    1356.53     42.56 o
 4   2  39      35.14     139.21     27.97 o
 5   2  39     229.81     261.04     30.15 o
 6   2  39     325.40     316.70     30.12 o
 7   2  39    1445.89    1436.78     46.54 o
 8   2  39      64.36     212.77     30.97 o
 9   2  39    1030.82    1002.04     43.30 o
10   2  39      79.04      71.17     35.22 o
 1   3  39     869.50    1009.45     46.15 o
 2   3  39     322.60     334.21     29.67 o
 3   3  39     357.80     356.83     31.10 o
 4   3  39     338.92     348.57     30.33 o
 5   3  39     353.14     340.58     32.22 o
 6   3  39    1620.29    1602.99     57.87 o
 7   3  39     325.03     312.58     32.26 o
 8   3  39    1151.38     990.31     41.70 o
 9   3  39     120.96     112.16     45.05 o
10   3  39       1.12      -8.56     35.31 o
 0   4  39   11452.58   10949.78    381.00 o
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 2   4  39    2846.48    2898.92     66.67 o
 3   4  39     292.37     274.92     31.62 o
 4   4  39     184.28     142.41     30.29 o
 5   4  39      32.35      66.92     29.87 o
 6   4  39    1044.01    1019.85     43.08 o
 7   4  39      84.45     151.07     50.81 o
 8   4  39     101.15     145.99     41.60 o
 9   4  39     629.96     595.22     39.36 o
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 2   5  39     528.14     554.79     49.59 o
 3   5  39      29.47      37.52     44.12 o
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 4   5  39    1207.06    1180.49    54.24 o
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 7   6  39     367.38     411.34    58.59 o
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 5   7  39       1.24      37.88    82.75 o
 6   7  39      26.81       4.74    51.45 o
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 2   0  40     905.24     946.14    52.22 o
 3   0  40     537.87     611.95    66.61 o
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 5   0  40     757.34     741.36    49.01 o
 6   0  40     313.97     279.81    42.79 o
 7   0  40     745.11     817.65    60.51 o
 8   0  40     227.83     193.75    45.14 o
 9   0  40     690.38     560.55    53.44 o
10   0  40     665.43     539.45    58.24 o
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 3   1  40     160.16     154.77    26.10 o
 4   1  40     467.91     402.44    29.94 o
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 8   1  40     541.31     556.83    44.29 o
 9   1  40       6.78      35.46    31.40 o
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 4   3  40    302.53     306.35     29.92 o
 5   3  40    173.07     185.91     29.92 o
 6   3  40    784.09     956.10     49.19 o
 7   3  40    272.98     282.99     32.00 o
 8   3  40    416.14     410.09     40.94 o
 9   3  40    238.10     288.36     36.18 o
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 7   4  40      1.63      58.06     43.53 o
 8   4  40     16.79      43.13     32.11 o
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 5   5  40    191.44     208.04     35.07 o
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 4   1  41      33.38      46.89     26.51 o
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 6   1  41       1.17     143.69     51.75 o
 7   1  41      66.61      57.27     29.97 o
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 2   2  41      24.40      78.42     27.75 o
 3   2  41     601.20     623.28     33.94 o
 4   2  41    1664.85    1575.43     46.55 o
 5   2  41     729.12     644.25     39.99 o
 6   2  41     141.70     142.03     29.79 o
 7   2  41      92.80     101.68     30.67 o
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 9   2  41     161.88     174.86     40.19 o
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 2   3  41     998.21     979.09     38.90 o
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 4   5  41     441.90     441.73     52.74 o
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 4   6  41      37.36      83.80     55.88 o
 5   6  41      28.91      43.10     54.52 o
 6   6  41      39.51       7.54     55.85 o
 7   6  41       7.12      27.73     59.68 o
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 2   7  41       3.58     -27.48     91.84 o
 3   7  41      10.35      64.71     79.05 o
 4   7  41       4.16     119.63    121.62 o
 5   7  41     266.84     237.39     60.02 o
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 3   0  42    1874.33    1704.42     67.27 o
 4   0  42     109.07      77.18     38.55 o
 5   0  42    1000.52    1006.97     53.65 o
 6   0  42     210.58     168.02     42.11 o
 7   0  42       1.29     -23.33     52.98 o
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 9   0  42       4.64     -44.90     44.90 o
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 2   1  42     222.64     274.49     24.23 o
 3   1  42     857.77     938.01     37.31 o
 4   1  42      41.52      71.25     27.33 o
 5   1  42     107.86     147.58     27.92 o
 6   1  42    1238.19    1226.17     43.67 o
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 8   1  42     777.52     826.86     41.36 o
 9   1  42      14.74      49.48     44.63 o
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 4   2  42     281.87     361.91     31.70 o
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 6   2  42     280.78     312.79     31.23 o
 7   2  42     342.37     360.29     34.56 o
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2  3  42    786.22    708.20    49.81 o
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4  3  42   1267.11   1215.79    43.57 o
5  3  42      5.43     78.98    32.62 o
6  3  42   1394.43   1259.04    65.46 o
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3  5  42    510.78    513.06    56.09 o
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6  5  42     67.12     44.28    37.30 o
7  5  42     38.26     11.61    43.89 o
8  5  42    124.39    121.38    35.39 o
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2  6  42    126.87    103.40    49.00 o
3  6  42      4.56    -26.27    46.71 o
4  6  42    217.65    258.19    53.50 o
5  6  42    212.15    189.21    57.17 o
6  6  42      8.61     -5.69    99.62 o
7  6  42      3.38    -26.07    72.23 o
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4  7  42      7.52     67.32    57.78 o
5  7  42     10.95     72.47    56.18 o
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7  1  43     38.97     32.24    29.23 o
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 4   2  43     158.55     220.52     31.30 o
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 6   2  43     232.97     172.51     30.89 o
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 5   6  43     105.09      46.77     53.44 o
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 3   7  43     130.30     166.21     60.59 o
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 6   0  44      47.23       50.94     41.57 o
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 3   4  44     378.35      441.64     34.64 o
 4   4  44      13.31       68.91     32.12 o
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 6   4  44      56.91      124.34     39.19 o
 7   4  44     524.82      463.00     44.58 o
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7  5  44    217.42    270.70    45.34 o
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1  6  44    571.86    533.13    56.39 o
2  6  44    179.98    161.83    49.19 o
3  6  44     77.45    159.82    54.69 o
4  6  44     10.99    -29.32    53.86 o
5  6  44     39.65     60.94    61.26 o
6  6  44    210.87    213.54    64.02 o
1  7  44     98.34     45.38    55.05 o
2  7  44      7.43     27.87    54.47 o
3  7  44    138.73    149.71    55.78 o
4  7  44      6.30     49.35    53.14 o
1  1  45   1113.55   1156.32    41.62 o
2  1  45     10.65     76.05    24.52 o
3  1  45    264.89    259.06    29.45 o
4  1  45    450.36    446.94    37.52 o
5  1  45     96.89    142.41    28.25 o
6  1  45      3.10     65.48    28.52 o
7  1  45     15.41     18.78    29.96 o
8  1  45      5.59     54.47    35.61 o
9  1  45    133.34    136.23    35.41 o
0  2  45    309.90   1062.16    84.91 o
1  2  45    311.34    335.90    35.83 o
2  2  45     34.11    111.46    29.36 o
3  2  45    241.03    258.14    32.50 o
4  2  45     76.16     87.40    29.24 o
5  2  45    535.09    507.62    42.02 o
6  2  45     43.52     47.40    30.42 o
7  2  45    413.49    380.49    34.53 o
8  2  45    185.04    207.24    39.42 o
9  2  45      9.19     49.76    34.45 o
1  3  45   2701.47   2668.89    74.74 o
2  3  45     58.77    113.92    30.06 o
3  3  45    164.22    218.89    31.42 o
4  3  45    249.62    259.89    31.06 o
5  3  45   1137.11    917.07    42.82 o
6  3  45    223.22    150.57    39.39 o
7  3  45   1072.10    991.15    44.66 o
8  3  45    427.07    423.45    38.48 o
9  3  45     12.26     25.98    37.18 o
0  4  45   2519.60   2518.24   125.80 o
1  4  45    126.78    203.44    47.86 o
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2  4  45      35.70      62.05     43.63 o
3  4  45    2286.08    2095.90     90.47 o
4  4  45     140.13     231.59     35.00 o
5  4  45      78.70     102.71     34.84 o
6  4  45     443.09     505.13     43.60 o
7  4  45     182.32     215.60     48.51 o
8  4  45     123.65     104.06     63.35 o
1  5  45       9.73       0.91     47.92 o
2  5  45     252.13     300.14     58.60 o
3  5  45     118.48      81.05     49.04 o
4  5  45      47.18      44.64     61.47 o
5  5  45      96.66     138.13     46.37 o
6  5  45      26.46      45.83     42.07 o
7  5  45     122.42     154.04     46.39 o
0  6  45     158.50     402.53     78.45 o
1  6  45     171.39     180.48     50.48 o
2  6  45     437.15     414.09     56.94 o
3  6  45     113.71     154.30     51.37 o
4  6  45      27.86     -12.30     57.92 o
5  6  45      25.70      95.78     71.99 o
6  6  45     164.22     167.14     65.46 o
1  7  45      30.97     101.89     55.64 o
2  7  45      21.27      -3.80     54.64 o
3  7  45      71.84      82.79     57.87 o
4  7  45       0.85      52.81     60.44 o
0  0  46    3813.34    3497.89    152.35 o
1  0  46      41.14     122.06     40.28 o
2  0  46    1273.15    1255.36     61.21 o
3  0  46     502.69     357.32     63.62 o
4  0  46      22.51      72.68     41.20 o
5  0  46    1393.53    1198.58     62.04 o
6  0  46    1397.10    1325.50     64.66 o
7  0  46    1578.75    1487.03    135.01 o
8  0  46       4.17       1.41     42.35 o
9  0  46       3.07     -42.34     49.28 o
1  1  46     872.75     814.86     38.60 o
2  1  46      11.98      47.45     28.03 o
3  1  46     108.81     143.31     32.17 o
4  1  46     375.53     348.87     52.92 o
5  1  46       4.41      41.63     26.87 o
6  1  46     501.06     486.90     33.88 o
7  1  46      45.77      32.48     30.78 o
8  1  46     204.48     207.60     34.41 o
9  1  46     237.80     294.65     38.33 o
0  2  46   12183.90   11144.53    223.56 o
1  2  46     746.02     753.21     40.25 o
2  2  46    1664.18    1498.87     47.81 o
3  2  46      36.94      29.77     28.36 o
4  2  46      31.01      85.17     29.84 o
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5  2  46     222.76     187.40     36.90 o
6  2  46     294.51     243.64     32.37 o
7  2  46     167.32     119.12     32.95 o
8  2  46     377.81     444.50     44.12 o
9  2  46     170.90     220.05     47.74 o
1  3  46    3821.06    3808.67     99.18 o
2  3  46     753.08     658.61     37.86 o
3  3  46      12.87      12.54     30.35 o
4  3  46      64.32     102.74     31.18 o
5  3  46     279.79     287.46     37.99 o
6  3  46      96.79     175.90     40.17 o
7  3  46      47.65     115.27     34.18 o
8  3  46     480.12     426.32     37.48 o
9  3  46      71.76      67.63     37.88 o
0  4  46    1469.00    1584.99    106.19 o
1  4  46     138.56     180.33     47.73 o
2  4  46     151.53     138.34     49.00 o
3  4  46       1.47      51.07     36.11 o
4  4  46     220.69     210.70     34.85 o
5  4  46      37.41      18.65     33.39 o
6  4  46     237.74     184.98     40.32 o
7  4  46     561.38     507.32     45.88 o
8  4  46      51.56      75.58     33.95 o
1  5  46      10.09       0.15     45.06 o
2  5  46       8.79     109.71     49.24 o
3  5  46     103.03     190.29     51.50 o
4  5  46     388.90     469.76     54.38 o
5  5  46     155.78     183.85     43.99 o
6  5  46     234.14     227.46     46.53 o
7  5  46     152.41     177.69     47.05 o
0  6  46    1234.78    1235.08     93.04 o
1  6  46      40.19      66.50     79.29 o
2  6  46     373.06     381.00     57.47 o
3  6  46      87.67      87.99     51.67 o
4  6  46       6.06     -25.01     52.40 o
5  6  46      89.20     102.29     59.73 o
1  7  46      13.18     -53.12     53.12 o
2  7  46      89.88      69.78     57.04 o
3  7  46     138.68     169.68     62.98 o
1  1  47      37.57      87.62     40.93 o
2  1  47      17.32      17.58     33.07 o
3  1  47     129.69     160.17     29.98 o
4  1  47       1.00     128.41     34.47 o
5  1  47      73.05      82.94     29.82 o
6  1  47     574.11     632.88     36.94 o
7  1  47     403.22     344.25     34.68 o
8  1  47     283.85     356.25     37.87 o
9  1  47      13.28      35.06     35.06 o
0  2  47    3493.50    3929.32     96.90 o
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4	2	47	61.20	28.93	39.29	o
5	2	47	0.05	55.92	35.81	o
6	2	47	732.45	642.56	37.93	o
7	2	47	4.41	43.49	44.29	o
8	2	47	322.77	347.71	38.23	o
9	2	47	305.44	391.45	51.33	o
1	3	47	797.86	775.63	44.50	o
2	3	47	129.69	127.28	30.64	o
3	3	47	331.71	388.38	43.36	o
4	3	47	75.08	77.53	30.85	o
5	3	47	3.95	29.45	37.14	o
6	3	47	689.07	618.28	45.42	o
7	3	47	119.70	159.15	36.30	o
8	3	47	338.12	263.98	36.84	o
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1	4	47	1161.16	1140.77	80.36	o
2	4	47	9.35	22.55	48.37	o
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4	4	47	11.37	10.37	32.73	o
5	4	47	441.99	396.68	38.25	o
6	4	47	414.82	426.02	44.26	o
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8	4	47	216.66	240.03	37.44	o
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3	5	47	882.44	823.81	87.78	o
4	5	47	73.14	31.93	51.40	o
5	5	47	0.42	56.52	50.26	o
6	5	47	263.32	280.03	48.21	o
7	5	47	20.36	23.10	85.37	o
0	6	47	7.39	41.62	71.27	o
1	6	47	90.17	118.16	51.37	o
2	6	47	23.74	34.74	50.13	o
3	6	47	288.93	324.36	77.37	o
4	6	47	42.54	30.32	59.83	o
5	6	47	1.78	74.10	60.36	o
1	7	47	339.82	247.41	60.14	o
2	7	47	306.91	369.03	81.08	o
3	7	47	127.75	184.09	66.85	o
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1	0	48	2872.85	2647.96	189.07	o
2	0	48	149.24	136.85	43.31	o
3	0	48	369.71	358.28	71.75	o
4	0	48	157.21	232.36	44.16	o
5	0	48	32.24	11.75	42.01	o
6	0	48	2.41	-15.11	38.47	o

7	0	48	0.00	-0.16	42.88	o
8	0	48	206.34	176.84	49.91	o
9	0	48	93.50	159.59	51.90	o
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5	1	48	70.42	68.12	31.12	o
6	1	48	943.65	867.78	40.09	o
7	1	48	28.77	41.18	32.80	o
8	1	48	4.48	-12.44	32.86	o
9	1	48	1.28	-14.77	35.87	o
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1	2	48	2318.68	2253.59	57.72	o
2	2	48	17.79	43.64	30.60	o
3	2	48	284.73	225.34	31.85	o
4	2	48	96.80	50.29	32.32	o
5	2	48	1202.51	933.67	53.65	o
6	2	48	53.04	15.89	30.25	o
7	2	48	40.04	37.84	33.50	o
8	2	48	224.61	207.34	37.57	o
9	2	48	60.21	125.76	36.95	o
1	3	48	37.63	64.25	41.99	o
2	3	48	203.71	208.16	33.65	o
3	3	48	44.37	98.62	30.15	o
4	3	48	57.52	73.74	31.71	o
5	3	48	311.11	306.52	39.52	o
6	3	48	245.82	351.33	43.69	o
7	3	48	202.68	302.21	36.40	o
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2	4	48	4.20	-19.18	45.58	o
3	4	48	354.69	349.11	46.16	o
4	4	48	524.57	560.08	39.48	o
5	4	48	51.51	7.42	35.62	o
6	4	48	362.50	395.66	43.21	o
7	4	48	38.53	70.54	42.61	o
1	5	48	1083.55	720.20	94.59	o
2	5	48	311.01	269.03	54.27	o
3	5	48	4.39	75.48	52.46	o
4	5	48	8.48	21.18	48.91	o
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3	6	48	116.10	179.04	63.62	o
4	6	48	29.31	30.85	61.49	o

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5  6  48     285.86     163.93     62.28 o
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2  7  48      46.07     127.12     62.64 o
1  1  49      77.20     245.30     31.83 o
2  1  49      6.41      -25.47     29.69 o
3  1  49     567.07     576.70     35.99 o
4  1  49     260.51     383.18     41.05 o
5  1  49      2.71      34.66     31.53 o
6  1  49     256.49     324.28     43.35 o
7  1  49      48.23      16.75     32.95 o
8  1  49      0.54      -13.99     35.29 o
9  1  49      35.00      52.68     37.00 o
0  2  49     238.65     411.46     98.06 o
1  2  49    1776.71    1691.23     51.13 o
2  2  49      36.58     135.80     31.53 o
3  2  49     254.50     235.79     31.92 o
4  2  49      2.26      16.17     32.78 o
5  2  49     273.33     382.20     43.46 o
6  2  49      0.46      36.84     32.96 o
7  2  49      39.50      69.87     33.48 o
8  2  49     289.78     193.12     57.79 o
1  3  49     324.10     405.88     59.20 o
2  3  49     132.54     179.70     33.06 o
3  3  49     556.65     580.23     38.65 o
4  3  49     359.24     502.57     36.92 o
5  3  49     188.55     172.29     39.64 o
6  3  49     138.38     160.40     40.13 o
7  3  49     124.55     181.14     36.54 o
8  3  49     143.88     136.85     36.46 o
0  4  49     272.23     355.89     70.32 o
1  4  49     578.34     525.73    102.49 o
2  4  49      90.51      51.23     50.29 o
3  4  49     337.68     294.92     51.99 o
4  4  49     218.81     272.26     36.90 o
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6  4  49      25.48      59.68     42.66 o
7  4  49     990.39    1030.07     60.84 o
1  5  49     108.57     195.77     55.29 o
2  5  49      45.75      68.56     48.58 o
3  5  49     128.36      80.68     55.13 o
4  5  49     698.60     758.22     64.59 o
5  5  49     843.13     878.71     69.48 o
6  5  49      25.18      -7.19     57.61 o
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1  6  49      15.80      -3.86     51.57 o
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3  6  49     265.31     250.89     60.88 o
4  6  49      11.97      32.78     68.88 o
1  7  49      8.54      -18.28     77.73 o
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2	0	50	2445.40	2217.28	83.11	o
3	0	50	522.39	570.04	48.01	o
4	0	50	748.92	705.77	51.35	o
5	0	50	111.54	91.36	45.41	o
6	0	50	66.90	41.33	43.46	o
7	0	50	33.11	53.10	48.88	o
8	0	50	285.24	333.90	92.08	o
9	0	50	257.22	301.27	56.51	o
1	1	50	2112.12	2052.26	56.54	o
2	1	50	354.81	384.95	34.69	o
3	1	50	456.69	460.08	54.79	o
4	1	50	2028.82	1741.89	61.17	o
5	1	50	646.58	635.45	48.09	o
6	1	50	28.39	68.85	30.93	o
7	1	50	5.13	52.51	32.80	o
8	1	50	7.34	37.49	34.29	o
0	2	50	152.28	213.98	45.11	o
1	2	50	123.80	369.04	34.95	o
2	2	50	2044.66	2038.38	57.59	o
3	2	50	7.93	0.11	31.70	o
4	2	50	923.43	952.70	50.55	o
5	2	50	531.66	472.03	41.71	o
6	2	50	11.55	51.85	42.98	o
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2	3	50	86.42	156.51	34.42	o
3	3	50	18.94	85.04	36.32	o
4	3	50	534.72	489.31	37.35	o
5	3	50	147.10	105.38	37.40	o
6	3	50	460.09	527.27	47.40	o
7	3	50	96.45	148.14	35.83	o
8	3	50	522.24	435.01	41.48	o
0	4	50	3.99	32.29	64.82	o
1	4	50	138.87	134.25	53.94	o
2	4	50	173.14	82.30	52.27	o
3	4	50	0.22	-12.11	65.77	o
4	4	50	37.22	25.27	40.86	o
5	4	50	50.77	14.14	36.00	o
6	4	50	22.91	65.65	40.16	o
7	4	50	140.03	311.01	47.06	o
1	5	50	41.61	75.19	82.51	o
2	5	50	201.85	235.20	73.31	o
3	5	50	90.41	68.43	56.44	o
4	5	50	291.80	340.64	56.32	o
5	5	50	50.59	118.55	57.52	o
6	5	50	4.59	38.29	55.64	o

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3  6 50     62.41     19.99     56.36 o
4  6 50     81.05    103.33     61.83 o
1  1 51      0.46    551.16     44.93 o
2  1 51    814.33    698.41     38.21 o
3  1 51     37.70     12.94     32.10 o
4  1 51    345.43    437.45     53.27 o
5  1 51    125.76    201.57     32.67 o
6  1 51    181.77    174.12     34.26 o
7  1 51      0.74     13.56     34.48 o
8  1 51      7.85     59.90     36.15 o
0  2 51   1867.38   2036.07    117.67 o
1  2 51   1817.85   1727.83     52.95 o
2  2 51     53.93    231.40     35.32 o
3  2 51     56.58     48.86     31.10 o
4  2 51    261.58    300.85     39.65 o
5  2 51     31.38     63.29     37.79 o
6  2 51    572.35    585.72     44.19 o
7  2 51    765.02    758.74     42.84 o
8  2 51     12.31     53.21     38.37 o
1  3 51     68.16     67.59     50.97 o
2  3 51    621.51    594.44     47.87 o
3  3 51     89.04    141.01     32.94 o
4  3 51     61.02    152.26     37.49 o
5  3 51    227.67    241.13     40.73 o
6  3 51    254.95    195.24     58.38 o
7  3 51      3.99     19.29     36.43 o
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1  4 51      4.15    -45.01     52.70 o
2  4 51     66.01     74.60     46.65 o
3  4 51    459.85    507.35    137.76 o
4  4 51     68.48    125.01     69.94 o
5  4 51     56.62     49.26     41.94 o
6  4 51     18.58     40.85     44.03 o
7  4 51    201.94    234.28     63.59 o
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2  5 51     40.82     97.42     73.55 o
3  5 51      0.07     -7.84     49.52 o
4  5 51      6.01     34.42     55.87 o
5  5 51     37.57     -4.12     52.95 o
0  6 51    178.66    224.34     79.41 o
1  6 51     76.61     46.73     53.42 o
2  6 51     10.32    -31.32     51.73 o
3  6 51      9.02     23.03     64.58 o
0  0 52   9096.02   8423.64    306.62 o
1  0 52   1140.69   1093.28     64.70 o
2  0 52   1132.44   1125.17     64.88 o
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4   0  52       0.00      48.00     45.89 o
5   0  52       4.27      25.68     45.96 o
6   0  52     141.24       58.79     49.75 o
7   0  52     595.67      526.27     59.25 o
8   0  52     132.49      135.59    100.21 o
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2   1  52     479.95      513.25     35.13 o
3   1  52      22.11       15.12     38.26 o
4   1  52       8.53      -15.91     36.01 o
5   1  52    1163.56     1158.75     46.36 o
6   1  52     754.21      703.83     41.87 o
7   1  52       1.47       23.85     34.77 o
8   1  52     236.84      264.43     39.37 o
0   2  52     660.26      937.32     82.51 o
1   2  52     164.70      223.51     33.37 o
2   2  52     776.74      700.42     39.57 o
3   2  52     517.41      482.71     39.46 o
4   2  52      61.35       70.37     36.50 o
5   2  52       7.74       42.73     39.17 o
6   2  52       5.31       19.98     34.44 o
7   2  52      92.83      165.35     37.50 o
8   2  52     202.44      265.76     39.26 o
1   3  52     387.58      336.07     49.80 o
2   3  52    1230.51     1282.14     58.19 o
3   3  52      20.72       55.50     34.23 o
4   3  52     558.87      524.37     40.51 o
5   3  52      22.11       44.25     40.44 o
6   3  52     154.66      141.93     43.57 o
7   3  52     118.77      284.67     39.76 o
0   4  52     320.74      335.32     75.10 o
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2   4  52      14.16       60.70     47.86 o
3   4  52      68.72       58.45     51.82 o
4   4  52      14.89      -12.46     41.73 o
5   4  52     108.37      100.16     44.24 o
6   4  52      10.46      -25.11     40.57 o
1   5  52      20.98       14.36     51.51 o
2   5  52     374.00      314.12     57.43 o
3   5  52       1.50      -12.22     51.05 o
4   5  52     226.22      211.70     57.04 o
5   5  52     233.93      177.72     63.48 o
0   6  52      96.21      126.52     78.45 o
1   6  52      54.77       78.31     92.44 o
2   6  52       0.76      -57.18     57.18 o
3   6  52       1.40       43.56     56.64 o
1   1  53    5133.90     4956.84    101.28 o
2   1  53      15.14       43.51     32.41 o
3   1  53       5.63      -12.93     36.01 o
```

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7   1  53      8.10     64.60    36.70 o
8   1  53     62.01     54.40    36.19 o
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3   2  53     24.23    120.24    35.29 o
4   2  53    135.58    184.86    63.53 o
5   2  53    131.22    164.09    42.68 o
6   2  53     65.57     35.22    35.70 o
7   2  53    329.66    385.21    39.95 o
1   3  53     92.72    110.09    50.27 o
2   3  53     39.22    129.41    39.79 o
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4   3  53     16.02     31.79    34.94 o
5   3  53     65.05     88.78    41.52 o
6   3  53      6.72     24.93    46.97 o
7   3  53     15.14     56.18    37.25 o
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1   4  53      4.03      8.73    48.24 o
2   4  53    480.66    401.60    60.57 o
3   4  53     36.66     13.24    73.55 o
4   4  53    354.36    443.17    48.78 o
5   4  53     29.36     44.24    42.00 o
6   4  53      0.67     11.40    45.13 o
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2   5  53     12.33     81.84    56.23 o
3   5  53     49.02     31.81    75.46 o
4   5  53    580.07    548.25    65.55 o
5   5  53     76.63     90.83    60.49 o
0   6  53    259.16    139.20    80.12 o
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0   0  54     68.85    258.31    73.90 o
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3   0  54   1799.92   1811.99    79.63 o
4   0  54   1750.18   1823.05    77.67 o
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7   0  54      9.48     16.98    48.93 o
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4   1  54      2.14     61.96    38.61 o
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6   1  54     83.33     69.76    38.96 o
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3  2  54    747.47    724.82    41.91 o
4  2  54    413.84    384.45    43.13 o
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6  2  54      2.14     31.92    34.31 o
7  2  54    112.59    163.02    38.30 o
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2  3  54    283.66    348.79    55.44 o
3  3  54     83.90    168.76    38.77 o
4  3  54      0.71     74.26    34.18 o
5  3  54     10.12     73.94    40.66 o
6  3  54    131.92    106.04    43.00 o
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2  4  54    126.38    158.63    58.54 o
3  4  54     69.03     49.56    50.46 o
4  4  54      5.76    105.66    51.24 o
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6  4  54     94.74     72.76    43.99 o
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4  5  54    610.49    843.35    91.36 o
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2  1  55     46.47      6.73    31.76 o
3  1  55    472.87    458.41    46.75 o
4  1  55    311.19    430.98    43.47 o
5  1  55     33.80     70.74    33.57 o
6  1  55     83.18     60.47    40.67 o
7  1  55    224.29    213.83    39.64 o
0  2  55      0.03     90.41    64.82 o
1  2  55   1192.34   1099.61    56.04 o
2  2  55   2152.48   2134.87    61.22 o
3  2  55    724.45    622.29    41.97 o
4  2  55    126.70    176.40    40.93 o
5  2  55     64.83    -17.94    41.67 o
6  2  55    260.52    260.95    38.79 o
7  2  55     32.18     40.41    36.83 o
1  3  55     23.64     44.81    48.69 o
2  3  55      3.62      4.47    47.24 o
3  3  55    298.04    284.12    40.58 o
4  3  55    745.10    753.04    59.58 o
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6  3  55    101.73    182.85    44.45 o
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1	3	57	698.10	668.15	67.45	o
2	3	57	785.77	796.88	66.77	o
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4	3	57	705.03	595.85	49.39	o
5	3	57	172.89	135.69	46.17	o
0	4	57	209.94	240.61	81.08	o
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0	2	59	2473.31	2160.68	132.02 o
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===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
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_shelx_F_calc_maximum      343.15
_exptl_crystal_F_000      1536.00
_reflns_d_resolution_high  0.7989

loop_
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 'x, y, z'
 '-x, y+1/2, -z+1/2'
 '-x, -y, -z'
 'x, -y-1/2, z-1/2'

_cell_length_a     8.5329
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_cell_angle_beta   96.534
_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
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 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
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```

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 5   0   0     776.55     560.86     86.29 o
 6   0   0       0.11     -29.52     94.23 o
 9   0   0     182.30     129.43     94.23 o
10   0   0      31.68       7.33     87.22 o
 2   1   0   55180.00   53076.96   2184.39 o
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 5   1   0     209.22     176.26     57.30 o
 6   1   0     988.74    1029.01     99.91 o
 8   1   0      93.07      86.29     79.47 o
 9   1   0      36.97      53.20     57.74 o
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 6   3   0    2938.03    2936.78     84.35 o
 7   3   0    1443.32    1250.32     71.27 o
 8   3   0      50.21     129.49     69.79 o
 9   3   0     141.02     209.44     60.56 o
10   3   0     114.76     146.49     83.38 o
 0   4   0   34395.43   35458.82   1698.46 o
 1   4   0    4406.63    4479.08    165.37 o
 2   4   0    5440.36    5595.93    107.20 o
 3   4   0       8.93      12.07     30.44 o
 4   4   0      11.62      -9.42     31.62 o
 5   4   0      81.92      70.40     42.89 o
 6   4   0      47.21     105.25     50.29 o
 7   4   0      29.96       6.48     57.29 o
 8   4   0      16.97      -2.63     76.51 o
 9   4   0       2.52     -71.69     99.05 o
10   4   0       0.03     -81.82     81.82 o
 1   5   0   25486.97   25683.64    875.46 o
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 3   5   0    5033.76    4833.70     95.31 o
 4   5   0      38.40      13.53     33.48 o
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 2   6   0    6653.92    6587.57    128.71 o
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7  11   0    1009.14    1014.09    79.48 o
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7  12   0     119.45      63.20    74.79 o
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9  12   0       0.17      -7.20    93.64 o
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4  14   0    2433.60    2470.20    93.08 o
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3	3	15	22.12	22.17	141.82	o
4	3	15	491.35	655.84	125.26	o
5	3	15	45.88	-77.62	88.47	o
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-6	4	15	185.93	95.38	77.84	o
-5	4	15	316.41	284.77	79.15	o
-4	4	15	0.45	13.70	76.84	o
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 3   3  19      4.25    101.05   282.70 o
-6   4  19     21.49    153.00   167.21 o
-5   4  19     71.45    -22.84   100.04 o
-4   4  19     19.31      6.17   116.21 o
```

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-3   4  19      1.33      45.90    151.05 o
-2   4  19    282.44     357.85    284.40 o
-1   4  19    466.50     504.59    166.62 o
 0   4  19    222.26     231.43    167.95 o
 1   4  19     89.20     172.09    176.59 o
 2   4  19    518.95     588.59    180.39 o
 3   4  19    714.30     298.23    187.64 o
-5   5  19      4.80     -61.26     99.07 o
-4   5  19     22.68     -96.82     96.82 o
-3   5  19      0.68     -10.32    113.59 o
-2   5  19     24.33     -93.80    151.42 o
-1   5  19     85.66     108.77    156.75 o
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 1   5  19    243.19     433.20    261.13 o
 2   5  19      0.57     -69.07    170.14 o
 3   5  19     67.00     -17.09    193.43 o
-5   6  19     58.53     146.45    103.70 o
-4   6  19      3.18     129.12    102.65 o
-3   6  19    143.05      72.01    113.18 o
-2   6  19     16.06      36.42    157.39 o
-1   6  19     77.88     -90.83    191.87 o
 0   6  19    127.59      39.71    264.53 o
 1   6  19      3.47    -162.74    162.74 o
 2   6  19    261.91      11.57    173.39 o
 3   6  19    169.71     199.72    177.00 o
-5   7  19      0.05    -136.22    146.00 o
-4   7  19    155.52     232.31    104.12 o
-3   7  19     12.66     -18.12    115.91 o
-2   7  19    209.26     244.10    170.33 o
-1   7  19     85.00    -150.43    150.43 o
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 1   7  19    848.82     939.26    212.88 o
 2   7  19     86.12     478.27    192.32 o
 3   7  19    118.17     287.03    187.81 o
-5   8  19      1.58       4.76    126.84 o
-4   8  19     22.91      98.97    113.22 o
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 3   8  19     32.45     309.76    214.58 o
-4   9  19    155.04     456.18    169.39 o
-3   9  19    101.56      -5.64    125.03 o
-2   9  19     33.27     -66.36    162.95 o
-1   9  19     60.12     189.58    277.02 o
 0   9  19    447.93     158.65    179.95 o
 1   9  19    253.66     362.11    185.62 o
```

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-4  10  19      31.38      75.87    167.31 o
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-2  10  19     276.07     426.55    353.09 o
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-3  11  19       1.31      40.35    214.50 o
-2  11  19      14.12     173.59    202.01 o
-1  11  19      34.73     -53.40    371.25 o
 0  11  19       6.34      46.98    198.89 o
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-3  12  19      49.11     169.84    225.01 o
-2  12  19     364.99     530.62    510.90 o
-1  12  19     182.27     529.08    214.27 o
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-2  13  19       6.55      51.93    221.60 o
-1  13  19       0.03     158.00    250.91 o
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-5   0  20     112.28      40.84    155.72 o
-4   0  20     151.81     196.51    162.92 o
-3   0  20      27.10      -5.68    258.86 o
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-1   0  20     540.37     526.80    290.65 o
 0   0  20     239.07     266.80    265.67 o
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-5   1  20       7.09      25.96    107.10 o
-4   1  20       4.92    -109.58    109.58 o
-3   1  20      93.08     -44.28    287.24 o
-2   1  20       0.23    -182.79    271.35 o
-1   1  20       1.03      18.17    275.89 o
 0   1  20      14.00    -281.56    281.56 o
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-5   3  20      10.14      47.03    108.50 o
-4   3  20     184.94      54.33    106.55 o
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-2   3  20      67.02      55.90    181.03 o
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-4   5  20     232.97     201.93    119.81 o
-3   5  20      17.42     413.63    203.79 o
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-2   6  20     227.50     181.52    170.36 o
-1   6  20     155.87      70.10    177.01 o
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 0   8  20      83.41     -70.16    294.05 o
 1   8  20       1.63    -134.92    281.00 o
-3   9  20      20.90      16.68    300.30 o
-2   9  20      80.70     -27.52    195.69 o
-1   9  20       1.81     195.63    194.43 o
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-2  10  20      41.64     346.52    273.62 o
-1  10  20      98.65     245.37    205.93 o
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-3   1  21     211.00     245.89    260.56 o
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 1   1  21    1122.30    1075.10    244.84 o
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   0   2  21     241.34     436.41    222.72 o
   1   2  21       4.20     -14.02    208.93 o
  -3   3  21       0.10     -11.98    188.19 o
  -2   3  21      32.72    -153.45    182.28 o
  -1   3  21       1.71     116.25    191.15 o
   0   3  21      58.51     -91.19    181.03 o
  -3   4  21      22.75    -200.95    200.95 o
  -2   4  21     203.70     369.04    184.12 o
  -1   4  21     314.18     528.99    177.96 o
   0   4  21     168.72     317.35    202.63 o
  -3   5  21     155.00      21.07    194.62 o
  -2   5  21      21.35      63.20    191.71 o
  -1   5  21       0.49    -175.38    175.38 o
   0   5  21       8.59     -65.51    179.50 o
  -2   6  21      51.77     127.92    656.22 o
  -1   6  21     259.59      13.11    193.39 o
   0   6  21      54.70    -241.83    241.83 o
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===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
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_shelx_F_calc_maximum      285.99
_exptl_crystal_F_000       818.00
_reflns_d_resolution_high  0.8394

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, -y, -z'

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_cell_length_b    11.8013
_cell_length_c    17.3963
_cell_angle_alpha  88.358
_cell_angle_beta   79.676
_cell_angle_gamma  83.259

_shelx_F_squared_multiplier     1.000

loop_
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_refln_index_k
_refln_index_l
_refln_F_squared_calc
_refln_F_squared_meas
_refln_F_squared_sigma
_refln_observed_status
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  5   0   0      59.38      50.77     2.19 o
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  8   0   0      74.95      88.24     4.76 o
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-10   1   0       3.78       4.52     5.14 o
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 -5   1   0    1295.28    1185.07    10.71 o
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 -1   1   0     544.18     230.67     2.29 o
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  3   1   0     133.87     139.02     2.22 o
  4   1   0       7.55       1.90     1.72 o
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 5   6   1     946.39    1058.50    17.18 o
 6   6   1     160.72     128.19     5.96 o
 7   6   1      47.01      47.29     3.41 o
 8   6   1     784.96     832.57    18.16 o
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-8   7   1     166.54     157.18    10.13 o
-7   7   1     564.04     533.28     8.81 o
-6   7   1       3.73       5.00    4.40 o
-5   7   1      20.33      19.93     3.67 o
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-3   7   1    1776.82    1666.22     11.35 o
-2   7   1     173.30     150.49      3.37 o
-1   7   1     287.82     271.90      5.34 o
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 3   7   1     510.66     478.25      3.86 o
 4   7   1    1793.33    1723.23     30.71 o
 5   7   1       4.93       7.20      3.67 o
 6   7   1      46.34      38.93      5.79 o
 7   7   1     182.41     175.11      6.21 o
 8   7   1      83.14      86.60      8.84 o
 9   7   1       0.50      -4.14      4.68 o
-7   8   1     256.66     248.21      6.80 o
-6   8   1     365.26     354.62      8.69 o
-5   8   1       2.40       6.31      3.03 o
-4   8   1      64.62      76.76      3.80 o
-3   8   1      16.89       8.43      2.90 o
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-7   9   1     248.34     247.58      7.07 o
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 5   9   1     187.65     172.64     10.81 o
 6   9   1     308.19     326.93      8.79 o
 7   9   1     103.56     113.54     10.50 o
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-6  10   1     171.47     174.71      6.22 o
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 5   2   2    3735.31    3801.20    27.59 o
 6   2   2     125.63     102.30     2.67 o
 7   2   2      44.49      39.43     3.38 o
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 6   3   2     252.37     235.82     3.89 o
 7   3   2      15.93      14.19     2.86 o
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 9   3   2     128.10     129.44     5.59 o
10   3   2      28.93      28.89     5.91 o
-9   4   2     217.66     232.27     7.25 o
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-7   4   2    1010.96    1020.64    19.23 o
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 7   4   2     649.04     701.18      9.82 o
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 8  -7   5       0.49       -0.26      5.01 o
-8  -6   5       1.98       -5.30      5.30 o
-7  -6   5     370.94      404.76       9.55 o
-6  -6   5     748.92      720.24      10.54 o
-5  -6   5     592.21      622.66      18.15 o
-4  -6   5     128.94      131.65       7.87 o
-3  -6   5     975.72      998.21      16.45 o
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 5  -6   5     319.19      318.96       4.19 o
 6  -6   5       1.24        0.81      2.43 o
 7  -6   5     493.57      498.92       8.54 o
 8  -6   5     301.28      311.20      13.44 o
-9  -5   5     114.82      123.63      12.15 o
-8  -5   5      46.50       27.01       5.72 o
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-2  -9   6      53.75      63.85     3.69 o
-1  -9   6     300.81     292.95     5.52 o
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 5  -9   6      49.14      50.61     3.98 o
 6  -9   6     262.54     277.19     6.87 o
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-7  -8   6     730.42     684.59    30.71 o
-6  -8   6     159.34     193.53    14.63 o
-5  -8   6     217.52     211.70    12.15 o
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 6  -8   6     122.38     122.92     4.99 o
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-8  -7   6      28.51      24.09     8.27 o
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-6  -7   6     641.84     577.78    23.68 o
-5  -7   6      12.86      15.59     3.87 o
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 3 -11   7     568.08     597.91    10.37 o
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-6  -8   7      39.91      44.47      4.85 o
-5  -8   7      69.47      67.65      4.89 o
-4  -8   7      41.11      37.72      5.95 o
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 5  10   7     122.26     124.18      4.75 o
 6  10   7      72.30      66.57      4.77 o
 7  10   7      16.05      18.32      4.40 o
-3  11   7       0.89      -2.79      4.00 o
-2  11   7      34.43      48.30      8.69 o
-1  11   7     264.10     281.89      8.38 o
 0  11   7     219.56     196.06      7.14 o
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 2  11   7     21.90     34.43     4.65 o
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 5  11   7      0.16     -3.21     4.55 o
 6  11   7    108.59    116.20     5.30 o
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-2  12   7    247.98    237.32     8.79 o
-1  12   7    127.08    154.49     7.14 o
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 2  12   7    209.02    195.13     7.03 o
 3  12   7     23.72     28.75     4.86 o
 4  12   7     49.39     43.84     4.96 o
 5  12   7    197.27    184.58     7.76 o
 0  13   7    232.24    222.84     7.76 o
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 3  13   7    281.39    252.62     7.96 o
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-2 -12   8     18.86     13.44     5.89 o
-1 -12   8    123.15    144.52     9.00 o
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 2 -12   8     65.21     63.98     5.09 o
 3 -12   8     76.73     91.06     8.94 o
-5 -11   8     27.18     52.08     7.39 o
-4 -11   8     10.88     11.18     5.27 o
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 6  -1  12     123.37     129.91      4.32 o
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3	-7	17	273.96	269.17	12.51 o
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-1	-6	17	1.13	-2.54	4.93 o
0	-6	17	57.96	58.68	5.32 o
1	-6	17	10.11	19.64	4.07 o
2	-6	17	15.53	19.01	4.30 o
3	-6	17	127.63	130.64	5.72 o
4	-6	17	156.43	149.92	10.44 o
-3	-5	17	0.18	0.77	5.06 o
-2	-5	17	540.36	549.80	12.09 o
-1	-5	17	142.30	146.59	5.77 o
0	-5	17	11.92	7.24	4.07 o
1	-5	17	8.78	13.20	3.84 o
2	-5	17	261.26	243.06	6.96 o
3	-5	17	256.26	243.60	6.71 o
4	-5	17	100.05	113.75	5.69 o

```
 5  -5  17       8.59      10.71      4.44 o
-3  -4  17      16.74      22.69      4.93 o
-2  -4  17       2.17       0.13      4.21 o
-1  -4  17     151.47     159.27      5.80 o
 0  -4  17       0.41       2.84      3.72 o
 1  -4  17     182.46     189.26      6.03 o
 2  -4  17     215.59     221.57      6.76 o
 3  -4  17      20.81       8.54      3.89 o
 4  -4  17       0.73       5.62      3.86 o
 5  -4  17     383.46     384.89      8.61 o
 6  -4  17     109.03      95.07      9.78 o
-4  -3  17     628.51     637.88     16.47 o
-3  -3  17     334.69     353.98      8.74 o
-2  -3  17       2.11      -3.92      3.92 o
-1  -3  17     236.70     236.14      7.06 o
 0  -3  17     838.56     878.00     32.42 o
 1  -3  17     338.18     350.18      7.71 o
 2  -3  17     119.06     126.01      6.45 o
 3  -3  17      91.53      81.74      4.93 o
 4  -3  17     119.03     102.83      5.15 o
 5  -3  17      46.78      42.27      4.99 o
 6  -3  17      27.77      32.17      4.70 o
-4  -2  17     453.03     428.12     10.04 o
-3  -2  17       3.10       5.75      4.50 o
-2  -2  17       4.32       0.51      4.17 o
-1  -2  17      16.01      16.99      4.32 o
 0  -2  17       5.24       7.74      4.09 o
 1  -2  17     725.47     741.14     16.75 o
 2  -2  17     367.66     351.03      7.74 o
 3  -2  17     119.26     129.61     10.59 o
 4  -2  17       0.28      -2.69      3.73 o
 5  -2  17      64.07      52.80      4.49 o
 6  -2  17     347.74     338.10     19.70 o
-4  -1  17      16.40      14.71      5.27 o
-3  -1  17     119.19     108.66      6.00 o
-2  -1  17     511.01     523.12      9.58 o
-1  -1  17      11.15       3.37      3.91 o
 0  -1  17     204.32     222.40      5.55 o
 1  -1  17       3.83      10.54      3.36 o
 2  -1  17      13.08      17.99      3.59 o
 3  -1  17      96.39      73.85      4.07 o
 4  -1  17    1157.89    1092.79     13.80 o
 5  -1  17     339.22     348.49      7.82 o
 6  -1  17      33.36      27.95      4.35 o
 7  -1  17       2.26       6.03      5.30 o
-4   0  17       0.78      -5.71      5.71 o
-3   0  17      10.03       5.98      4.31 o
-2   0  17     119.64     128.62      5.17 o
-1   0  17      74.76      77.60      4.60 o
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```
 0   0  17      65.26      68.92     4.05 o
 1   0  17      60.70      55.75     3.90 o
 2   0  17     445.34     437.95     7.36 o
 3   0  17    1204.81    1166.24    11.43 o
 4   0  17     941.82     898.84    10.17 o
 5   0  17       4.42       4.28     3.62 o
 6   0  17     323.10     337.99     8.14 o
 7   0  17     158.69     158.20     6.52 o
-4   1  17     128.55     115.20     8.95 o
-3   1  17     216.30     229.04     9.09 o
-2   1  17      54.84      59.72     5.21 o
-1   1  17     283.75     305.33     7.64 o
 0   1  17      17.85      25.25     4.00 o
 1   1  17     190.81     203.20     5.53 o
 2   1  17     740.15     794.31     9.45 o
 3   1  17      79.47      92.37    10.71 o
 4   1  17      99.31      98.37     6.14 o
 5   1  17     460.85     454.37     9.01 o
 6   1  17     245.08     227.05     6.80 o
 7   1  17       7.20      17.35     4.55 o
-4   2  17      61.57      51.08     5.49 o
-3   2  17      88.31      85.09     6.09 o
-2   2  17      28.31      32.93     4.61 o
-1   2  17     412.98     417.62    13.62 o
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 2   2  17       0.18       4.87     3.49 o
 3   2  17     187.14     190.41    15.13 o
 4   2  17      80.54      78.21     4.62 o
 5   2  17     116.41     122.17     5.43 o
 6   2  17       1.08       5.70     4.31 o
 7   2  17     214.18     190.53    10.64 o
-3   3  17       8.17      13.42     5.31 o
-2   3  17      40.38      54.10     5.85 o
-1   3  17      54.48      71.42     5.39 o
 0   3  17       1.19       4.46     5.21 o
 1   3  17      27.88      24.90     4.29 o
 2   3  17      10.24      12.45     3.55 o
 3   3  17     116.13     113.93     5.13 o
 4   3  17      44.67      37.81     4.22 o
 5   3  17      54.80      55.84     4.74 o
 6   3  17     311.82     303.21     7.56 o
 7   3  17     101.50      88.54     7.23 o
-3   4  17     113.01     128.87     6.58 o
-2   4  17     122.46     120.93     5.64 o
-1   4  17      46.87      58.80     5.64 o
 0   4  17     142.73     146.42     5.85 o
 1   4  17     395.41     414.35    12.80 o
 2   4  17       2.50       8.42     3.65 o
```

```
 3   4  17      98.00      91.96     4.85 o
 4   4  17     202.44     183.81     6.21 o
 5   4  17     126.14     118.42     5.53 o
 6   4  17       3.72       2.80    5.56 o
 7   4  17       0.54      -0.33    4.29 o
-2   5  17     145.22     159.61     6.58 o
-1   5  17      43.84      61.81     5.12 o
 0   5  17      45.67      48.51     4.56 o
 1   5  17      60.12      40.07     4.18 o
 2   5  17     170.41     157.90     8.48 o
 3   5  17     476.65     490.47     8.87 o
 4   5  17     248.55     248.83     6.62 o
 5   5  17      28.34      35.84     4.84 o
 6   5  17      37.61      37.26     4.74 o
-2   6  17      72.52      74.96     5.66 o
-1   6  17       3.33      -0.56    4.01 o
 0   6  17     109.36     113.74     5.59 o
 1   6  17     119.82     145.38     5.62 o
 2   6  17      85.79     103.71     6.41 o
 3   6  17       0.11      -2.18    3.67 o
 4   6  17     106.25      98.53     5.21 o
 5   6  17     337.22     364.32    13.58 o
 6   6  17      49.73      37.06     4.87 o
-1   7  17      49.01      37.85     8.38 o
 0   7  17     299.60     346.14    14.74 o
 1   7  17      17.51       9.66     5.32 o
 2   7  17       0.22       6.83     4.82 o
 3   7  17     158.31     169.39     7.52 o
 4   7  17     184.52     167.90    13.60 o
 5   7  17      10.77      13.75     7.55 o
 1   8  17     109.74      96.80     6.65 o
 2   8  17     455.40     464.96    10.97 o
 3   8  17      96.96      93.22     6.67 o
 0  -6  18      29.68      30.03     4.86 o
 1  -6  18       3.43       0.93     5.17 o
 2  -6  18       0.10      -4.23     4.23 o
 3  -6  18     388.92     375.95    10.84 o
-2  -5  18     561.36     599.33    10.89 o
-1  -5  18      15.34      23.96     4.92 o
 0  -5  18      16.76      18.57     4.10 o
 1  -5  18     150.46     155.69     5.89 o
 2  -5  18     543.28     545.67    10.50 o
 3  -5  18      29.46      21.03     4.25 o
 4  -5  18       0.42       2.66     4.45 o
-2  -4  18      63.78      70.63     5.55 o
-1  -4  18     256.49     252.69     7.40 o
 0  -4  18     340.07     330.45    12.15 o
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 2  -4  18      39.77      50.91     4.75 o
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 3  -4  18      16.81      8.09     3.99 o
 4  -4  18     212.99    186.74     6.57 o
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-2  -3  18      52.38     57.83     5.29 o
-1  -3  18     152.68    158.32     6.31 o
 0  -3  18     161.23    170.62     9.16 o
 1  -3  18       0.55      6.90    4.16 o
 2  -3  18     360.90    375.23     8.17 o
 3  -3  18      44.76     48.90     4.61 o
 4  -3  18     167.49    160.94     6.17 o
 5  -3  18      32.59     35.95     4.77 o
-3  -2  18      27.48     40.43     5.46 o
-2  -2  18      36.90     33.38     5.17 o
-1  -2  18     114.13     97.51     5.69 o
 0  -2  18     178.26    189.35     8.94 o
 1  -2  18    1212.15   1198.97    20.78 o
 2  -2  18     150.31    138.31     5.70 o
 3  -2  18       3.25     -3.87    4.14 o
 4  -2  18     137.04    130.48     5.69 o
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 6  -2  18      81.06     76.32     5.50 o
-3  -1  18     118.36    114.84     6.13 o
-2  -1  18      19.72     15.70     4.95 o
-1  -1  18      34.57     41.39     4.99 o
 0  -1  18      47.19     48.99     4.94 o
 1  -1  18       0.52     -4.19    4.19 o
 2  -1  18     285.12    290.19     7.28 o
 3  -1  18     515.56    465.82    11.39 o
 4  -1  18     350.05    304.60     8.17 o
 5  -1  18      88.38     90.41     5.29 o
 6  -1  18      37.22     31.05     5.02 o
-3   0  18      29.80     45.32     6.80 o
-2   0  18       1.94     -0.33    3.69 o
-1   0  18     137.48    146.37     5.70 o
 0   0  18      13.05     13.16     3.59 o
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 4   0  18      54.42     58.72     4.22 o
 5   0  18     712.17    651.93    10.73 o
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 2   1  18       7.92      9.49    4.06 o
 3   1  18       1.19      0.37    3.88 o
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 4   1  18     479.35     477.36     9.09 o
 5   1  18     600.39     563.80    16.20 o
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-3   2  18      10.59       6.69     7.10 o
-2   2  18     183.69     175.65     9.18 o
-1   2  18     349.08     370.18    15.30 o
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 3   2  18     570.22     578.68    18.47 o
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 3   3  18      44.74      42.46     4.63 o
 4   3  18      16.16      16.38     3.78 o
 5   3  18      68.28      60.31     7.10 o
 6   3  18      92.92     104.51     5.87 o
-2   4  18      14.21      28.40     5.80 o
-1   4  18      53.40      58.25     5.39 o
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 4   4  18     287.54     299.41     7.53 o
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 4   5  18       8.29      11.77     5.47 o
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 2   6  18       6.34       9.44     4.15 o
 3   6  18      89.60      92.42     5.08 o
 4   6  18     347.44     345.98     8.43 o
 5   6  18     149.29     161.73     6.09 o
 1   7  18       4.93      -7.14     7.14 o
 2   7  18     216.33     237.84    12.20 o
 3   7  18     143.78     142.29    10.24 o
 1  -5  19      21.29      26.85     4.75 o
 2  -5  19      85.49      84.46     5.57 o
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2	-4	19	231.42	231.58	12.27	o
3	-4	19	61.99	58.54	4.94	o
-1	-3	19	3.17	2.18	4.13	o
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-1	-2	19	35.99	53.54	5.25	o
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1	-2	19	550.79	591.27	10.37	o
2	-2	19	1.24	9.46	4.42	o
3	-2	19	51.49	54.85	4.73	o
4	-2	19	238.25	245.66	7.46	o
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-2	-1	19	15.10	16.29	4.59	o
-1	-1	19	307.69	329.31	8.41	o
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2	-1	19	11.73	3.96	4.16	o
3	-1	19	415.69	400.68	10.84	o
4	-1	19	67.11	53.96	5.09	o
5	-1	19	146.83	124.91	5.80	o
-2	0	19	18.52	18.71	5.58	o
-1	0	19	14.15	14.10	4.36	o
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1	0	19	269.84	291.19	11.16	o
2	0	19	8.37	9.11	3.62	o
3	0	19	22.98	30.24	3.98	o
4	0	19	145.06	131.01	6.00	o
5	0	19	479.05	445.59	10.70	o
-2	1	19	43.22	42.94	5.20	o
-1	1	19	86.01	88.15	5.42	o
0	1	19	239.07	250.26	7.46	o
1	1	19	0.20	1.55	4.24	o
2	1	19	0.73	2.74	4.26	o
3	1	19	436.37	445.62	9.15	o
4	1	19	358.00	353.11	9.68	o
5	1	19	0.27	-4.14	4.29	o
-2	2	19	152.11	148.52	8.35	o
-1	2	19	48.43	31.43	4.97	o
0	2	19	240.56	272.94	10.81	o
1	2	19	239.85	249.43	9.24	o
2	2	19	670.66	710.09	22.64	o
3	2	19	118.32	120.02	8.00	o

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 5   2  19    171.11    213.39     7.26 o
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 1   3  19    368.15    376.46     8.55 o
 2   3  19    149.68    156.98     8.77 o
 3   3  19     60.40     70.72     4.87 o
 4   3  19    273.41    275.71     7.46 o
 5   3  19    142.42    156.91     6.51 o
-1   4  19    281.21    288.77     7.89 o
 0   4  19     15.14     19.30     4.26 o
 1   4  19      0.81      9.17     4.03 o
 2   4  19    119.49    135.15     5.85 o
 3   4  19    281.27    307.06    10.30 o
 4   4  19     22.23     25.23     4.51 o
 5   4  19     20.48     18.79     4.69 o
 0   5  19     29.35     36.05     5.07 o
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 3   5  19      4.69     10.86     3.98 o
 4   5  19     87.48     95.65     5.42 o
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 2  -2  20    112.72    125.15     6.24 o
 3  -2  20    243.16    248.09     8.62 o
 0  -1  20      1.25      1.39     5.01 o
 1  -1  20    147.52    165.79     6.43 o
 2  -1  20     64.73     76.87     5.61 o
 3  -1  20     31.57     28.59     6.42 o
 4  -1  20      0.77      2.18     5.39 o
 0   0  20    264.19    243.90    11.32 o
 1   0  20      7.93     13.11     4.89 o
 2   0  20      3.69     19.33     4.31 o
 3   0  20     62.64     71.90     5.44 o
 4   0  20    340.26    347.62    10.31 o
 0   1  20     40.87     42.50     6.61 o
 1   1  20    231.63    274.20    15.42 o
 2   1  20    216.92    217.19     8.80 o
 3   1  20    125.01    114.49     6.11 o
 4   1  20     34.34     51.10     4.90 o
 0   2  20     51.98     40.72     5.28 o
 1   2  20     42.27     49.48     9.03 o
 2   2  20    207.54    228.27     7.30 o
 3   2  20     75.01     70.66     6.78 o
 4   2  20    534.71    546.59    10.26 o
 1   3  20      0.29     -0.01     4.60 o
 2   3  20    136.61    134.14     5.91 o
```

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   3   3  20      319.51      333.23      8.29 o
   2   4  20      228.22      235.46      7.44 o

===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
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_shelx_refln_list_code          4
_shelx_F_calc_maximum       257.70
_exptl_crystal_F_000        764.00
_reflns_d_resolution_high  0.7972

loop_
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 'x, y, z'
 '-x, -y, -z'

_cell_length_a     8.3507
_cell_length_b    11.1461
_cell_length_c    18.1235
_cell_angle_alpha  86.826
_cell_angle_beta   82.396
_cell_angle_gamma  85.480

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
 _refln_F_squared_meas
 _refln_F_squared_sigma
 _refln_observed_status
   2   0   0     1377.22     1441.02     46.39 o
   3   0   0       74.49      110.90      7.97 o
   4   0   0     6007.64     6172.61    116.93 o
   5   0   0       21.68       -7.74      7.74 o
   6   0   0      520.59      530.26     13.83 o
   7   0   0       58.11       59.44     15.95 o
   8   0   0       48.08       55.09     18.85 o
   9   0   0       50.92       65.24     21.02 o
  10   0   0      186.94      108.73     24.65 o
 -10   1   0       16.85       -1.45     23.92 o
  -9   1   0      932.49      955.36     38.42 o
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4	1	0	36.33	39.05	5.29 o
5	1	0	29.55	7.76	5.46 o
6	1	0	54.80	47.84	13.05 o
7	1	0	454.77	471.16	22.47 o
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3	10	7	8.91	3.14	7.11	o
4	10	7	722.25	715.56	16.56	o
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6	10	7	212.32	197.60	13.33	o
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5	11	7	568.07	546.01	15.02	o
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3	12	7	135.77	131.04	11.01	o
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 6  -7  13       52.13       37.71     13.73 o
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3	8	15	408.09	396.54	16.40	o
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 7   5  17      27.82      41.32     24.65 o
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3	-1	20	803.93	843.73	37.69	o
4	-1	20	131.24	133.37	27.54	o
5	-1	20	64.31	59.44	28.27	o
-3	0	20	71.39	36.97	26.82	o
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2	0	20	1331.33	1506.25	55.09	o
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4	0	20	140.20	129.75	26.82	o
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4	1	20	240.08	297.19	28.27	o
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2	3	20	186.85	168.17	23.92	o

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 3  -1  21     500.25     544.37     31.89 o
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 0   0  21     164.33     219.63     27.54 o
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   2   3  22      469.24      500.15     31.89 o
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   2   4  22        5.68      -20.30     25.37 o

===END of fcf

===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
#
data_[Fe(tpt)Cl2]2(µ-O).2(H2O), 6.4

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_shelx_F_calc_maximum      536.74
_exptl_crystal_F_000      1880.00
_reflns_d_resolution_high  0.7985

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 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, y+1/2, -z+1/2'
 '-x, -y, -z'
 'x, -y-1/2, z-1/2'

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_cell_length_c    22.7469
_cell_angle_alpha  90.000
_cell_angle_beta   93.135
_cell_angle_gamma  90.000

_shelx_F_squared_multiplier     1.000

loop_
 _refln_index_h
 _refln_index_k
 _refln_index_l
 _refln_F_squared_calc
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_refln_F_squared_sigma
_refln_observed_status
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 6   0   0    13155.09    10982.54    255.07 o
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 8   0   0      304.96      292.22     52.85 o
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 9   1   0       17.41       14.96     27.37 o
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11   1   0      475.66      398.28     64.50 o
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-7   3  24     382.78     414.69     62.23 o
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===END of fcf

#
# h,k,l, Fc-squared, Fo-squared, sigma(Fo-squared) and status flag
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_shelx_F_calc_maximum      124.03
_exptl_crystal_F_000       302.00
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 'x, y, z'
 '-x, -y, -z'

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_cell_length_c    11.4655
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_cell_angle_beta   98.399
_cell_angle_gamma 101.504

_shelx_F_squared_multiplier     1.000

loop_
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 _refln_index_k
 _refln_index_l
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 _refln_F_squared_meas
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-4	7	2	1.30	1.66	1.33 o
-3	7	2	917.57	943.36	41.24 o
-2	7	2	365.52	372.51	18.94 o
-1	7	2	751.40	680.40	25.49 o
0	7	2	425.17	408.43	10.77 o
-3	8	2	651.22	661.07	17.28 o
-2	8	2	1.20	0.60	1.37 o
-1	8	2	681.45	654.05	24.63 o
4	-8	3	0.08	1.28	1.03 o
-3	-7	3	106.42	119.51	4.82 o
-2	-7	3	346.20	350.45	12.92 o
3	-7	3	16.20	20.32	1.55 o
4	-7	3	814.27	837.77	20.73 o
5	-7	3	6.26	16.33	1.40 o
-4	-6	3	23.69	26.52	2.07 o
-3	-6	3	0.94	1.72	1.55 o
-2	-6	3	938.69	1029.13	37.20 o
0	-6	3	187.20	189.78	7.41 o
1	-6	3	4059.45	4001.47	147.07 o
3	-6	3	878.38	856.40	46.32 o
4	-6	3	667.39	761.33	18.90 o
5	-6	3	52.14	62.21	2.32 o
-4	-5	3	4.57	4.99	1.55 o
-3	-5	3	494.20	522.48	19.12 o
-2	-5	3	175.54	186.85	6.89 o
0	-5	3	307.02	235.41	8.95 o
1	-5	3	1221.50	1248.69	45.98 o
2	-5	3	664.26	604.79	16.79 o
3	-5	3	2601.32	2519.96	62.00 o
4	-5	3	507.82	523.50	18.00 o
5	-5	3	9.82	9.99	1.32 o
6	-5	3	949.10	958.00	57.17 o
-5	-4	3	0.34	-1.57	1.57 o
-4	-4	3	52.77	55.37	1.95 o
-3	-4	3	1829.24	1855.71	45.72 o
-2	-4	3	23.37	23.40	1.03 o

-1	-4	3	2535.11	2737.83	67.41 o
0	-4	3	1401.84	1327.91	48.91 o
1	-4	3	74.86	83.35	3.27 o
2	-4	3	33.88	79.99	2.21 o
3	-4	3	1002.85	1073.29	21.41 o
4	-4	3	12.81	8.55	0.88 o
5	-4	3	1399.47	1445.19	28.83 o
6	-4	3	289.56	292.24	10.33 o
-5	-3	3	990.76	953.72	21.48 o
-4	-3	3	3343.27	3306.73	65.76 o
-3	-3	3	9.25	9.87	0.91 o
-2	-3	3	4790.39	5732.09	140.90 o
-1	-3	3	253.28	173.20	4.91 o
0	-3	3	178.23	175.65	6.03 o
1	-3	3	592.26	411.37	9.20 o
2	-3	3	953.30	935.58	18.67 o
3	-3	3	108.76	128.68	2.75 o
4	-3	3	409.43	405.21	8.21 o
5	-3	3	83.46	90.92	2.21 o
6	-3	3	339.17	337.36	11.88 o
-6	-2	3	495.15	424.50	14.81 o
-5	-2	3	191.52	193.56	7.06 o
-4	-2	3	11.77	10.58	0.88 o
-3	-2	3	2413.55	2659.60	52.89 o
-2	-2	3	2862.47	3260.40	64.80 o
-1	-2	3	6.17	7.12	0.40 o
0	-2	3	4454.56	4596.76	144.02 o
1	-2	3	152.48	110.60	5.20 o
2	-2	3	58.75	84.74	1.86 o
3	-2	3	1589.90	1551.93	31.54 o
4	-2	3	6.38	5.58	0.88 o
5	-2	3	227.93	245.23	8.61 o
6	-2	3	38.84	35.99	2.58 o
-6	-1	3	594.72	527.82	18.25 o
-5	-1	3	80.82	75.77	3.10 o
-4	-1	3	192.60	204.00	4.27 o
-3	-1	3	143.22	115.77	2.45 o
-2	-1	3	5.41	3.38	0.52 o
-1	-1	3	88.13	144.73	2.95 o
0	-1	3	3345.99	3165.73	106.77 o
1	-1	3	43.48	57.16	1.50 o
2	-1	3	1973.12	1921.32	38.19 o
3	-1	3	1936.15	1978.21	39.35 o
4	-1	3	137.66	157.74	5.68 o
5	-1	3	861.87	858.64	29.28 o
6	-1	3	95.07	101.43	4.31 o
-6	0	3	179.57	166.18	6.20 o
-5	0	3	21.11	20.15	1.72 o
-4	0	3	53.01	37.03	1.89 o

-3	0	3	350.74	355.86	6.28 o
-2	0	3	845.16	1124.70	38.06 o
-1	0	3	5670.79	6040.95	203.55 o
0	0	3	423.92	393.86	12.49 o
1	0	3	420.60	399.70	13.60 o
2	0	3	2008.61	1979.47	40.24 o
3	0	3	25.84	37.68	2.76 o
4	0	3	1241.13	1325.16	44.95 o
5	0	3	1532.67	1515.28	51.32 o
6	0	3	99.25	93.85	4.13 o
-6	1	3	90.22	82.66	3.44 o
-5	1	3	1388.06	1430.20	48.39 o
-4	1	3	801.21	786.44	22.99 o
-3	1	3	27.97	12.72	0.78 o
-2	1	3	1627.10	1488.40	36.59 o
-1	1	3	800.61	816.15	20.12 o
0	1	3	7123.06	6637.70	135.54 o
1	1	3	51.30	14.11	1.30 o
2	1	3	273.49	282.71	7.05 o
3	1	3	161.77	187.02	5.77 o
4	1	3	1955.72	1922.72	64.92 o
5	1	3	27.51	26.00	2.07 o
-6	2	3	350.58	362.67	12.74 o
-5	2	3	28.30	31.58	1.45 o
-4	2	3	727.63	767.38	18.97 o
-3	2	3	1980.48	2072.59	51.06 o
-2	2	3	2630.52	2615.18	64.37 o
-1	2	3	1199.30	1220.29	25.00 o
0	2	3	1453.77	1180.56	28.07 o
1	2	3	841.43	787.50	16.17 o
2	2	3	379.06	375.73	9.42 o
3	2	3	657.15	634.90	15.74 o
4	2	3	184.96	184.52	4.87 o
5	2	3	40.63	36.51	2.41 o
-6	3	3	362.00	362.33	12.57 o
-5	3	3	23.39	19.32	1.20 o
-4	3	3	827.25	814.45	20.12 o
-3	3	3	436.81	480.52	11.92 o
-2	3	3	100.12	117.78	3.10 o
-1	3	3	6162.41	6424.90	131.33 o
0	3	3	458.09	379.80	7.84 o
1	3	3	105.10	100.55	2.86 o
2	3	3	1678.57	1623.45	40.00 o
3	3	3	645.11	645.81	16.11 o
4	3	3	198.46	190.27	6.20 o
-6	4	3	1.47	0.98	1.27 o
-5	4	3	1409.07	1377.78	34.04 o
-4	4	3	344.07	369.50	9.30 o
-3	4	3	181.73	196.87	4.99 o

-2	4	3	482.66	469.51	12.23 o
-1	4	3	835.76	826.03	21.36 o
0	4	3	192.16	190.89	5.11 o
1	4	3	3248.77	3033.29	78.04 o
2	4	3	110.97	98.68	3.96 o
3	4	3	632.29	603.94	22.04 o
4	4	3	273.81	249.02	9.47 o
-6	5	3	539.78	538.16	19.80 o
-5	5	3	911.34	912.88	33.06 o
-4	5	3	31.88	27.21	1.72 o
-3	5	3	91.12	89.04	2.72 o
-2	5	3	628.04	600.15	15.58 o
-1	5	3	93.02	90.21	2.60 o
0	5	3	185.13	171.91	4.74 o
1	5	3	343.77	326.56	8.70 o
2	5	3	6.09	8.27	1.38 o
3	5	3	34.23	39.61	2.58 o
-5	6	3	42.09	44.95	2.58 o
-4	6	3	350.14	387.98	13.60 o
-3	6	3	411.38	438.52	11.50 o
-2	6	3	221.49	208.49	5.65 o
-1	6	3	26.89	21.33	1.30 o
0	6	3	544.64	523.96	13.70 o
1	6	3	0.15	0.34	1.55 o
-4	7	3	227.70	254.15	6.93 o
-3	7	3	164.24	172.35	6.63 o
-2	7	3	786.62	826.46	21.49 o
-1	7	3	304.92	308.02	8.21 o
0	7	3	118.43	113.18	3.70 o
3	-8	4	4.14	6.20	1.21 o
4	-8	4	89.65	102.95	2.91 o
-3	-7	4	68.14	77.49	3.44 o
-2	-7	4	524.69	554.17	20.15 o
4	-7	4	420.72	425.91	10.70 o
5	-7	4	103.12	87.71	2.93 o
-4	-6	4	15.03	17.41	1.57 o
-3	-6	4	17.09	10.16	1.55 o
-2	-6	4	12.62	12.40	1.38 o
3	-6	4	1223.65	1207.60	29.79 o
4	-6	4	0.70	1.03	1.10 o
5	-6	4	695.43	750.84	26.61 o
-4	-5	4	1083.26	1136.93	28.15 o
-3	-5	4	4.41	5.51	1.38 o
-2	-5	4	271.76	305.67	11.19 o
-1	-5	4	12.35	20.32	1.21 o
2	-5	4	782.05	565.36	13.98 o
3	-5	4	91.68	97.55	3.01 o
4	-5	4	570.99	609.34	15.20 o
5	-5	4	772.83	827.53	42.62 o

-5	-4	4	1597.63	1551.74	38.42	o
-4	-4	4	3.45	2.34	1.02	o
-3	-4	4	1665.80	1770.68	35.18	o
-2	-4	4	1112.70	1179.75	23.46	o
-1	-4	4	582.02	650.86	13.54	o
0	-4	4	207.10	166.18	5.68	o
1	-4	4	1695.84	1551.18	32.51	o
2	-4	4	204.38	181.29	7.24	o
3	-4	4	2181.84	2191.76	43.48	o
4	-4	4	241.65	229.71	4.77	o
5	-4	4	1.61	3.74	1.07	o
6	-4	4	139.06	145.17	5.68	o
-5	-3	4	598.00	593.17	11.98	o
-4	-3	4	211.93	211.41	4.44	o
-3	-3	4	1392.09	1503.67	29.95	o
-2	-3	4	1825.80	2049.18	40.71	o
-1	-3	4	57.44	46.25	1.06	o
0	-3	4	15384.27	17091.14	420.45	o
1	-3	4	369.69	340.80	6.82	o
2	-3	4	1203.66	1223.56	24.39	o
3	-3	4	658.59	666.54	13.33	o
4	-3	4	116.83	117.46	2.64	o
5	-3	4	1.73	3.33	1.46	o
6	-3	4	311.58	347.00	12.23	o
-6	-2	4	593.15	546.42	18.94	o
-5	-2	4	0.64	2.07	1.55	o
-4	-2	4	587.01	555.36	11.18	o
-3	-2	4	2139.50	2350.97	46.76	o
-2	-2	4	118.73	149.20	3.08	o
-1	-2	4	5970.80	6265.90	124.50	o
0	-2	4	3444.48	3556.56	123.29	o
1	-2	4	0.84	4.32	0.61	o
2	-2	4	5407.58	5445.28	108.15	o
3	-2	4	179.47	159.82	3.41	o
4	-2	4	336.07	356.98	7.47	o
5	-2	4	468.79	482.02	16.53	o
6	-2	4	155.19	159.47	6.20	o
-6	-1	4	0.61	0.69	1.89	o
-5	-1	4	451.19	434.14	14.98	o
-4	-1	4	906.50	906.69	18.17	o
-3	-1	4	175.99	204.30	4.20	o
-2	-1	4	467.46	349.98	7.01	o
-1	-1	4	117.85	91.89	1.92	o
0	-1	4	2263.60	2281.99	39.93	o
1	-1	4	29.42	57.06	2.84	o
2	-1	4	1352.06	1292.48	29.05	o
3	-1	4	3.90	9.20	1.40	o
4	-1	4	306.76	319.45	11.02	o
6	-1	4	29.83	33.24	2.58	o

-6	0	4	226.48	220.77	7.92 o
-5	0	4	471.47	457.91	15.84 o
-4	0	4	3.12	4.65	1.38 o
-3	0	4	3724.55	3814.20	66.23 o
-2	0	4	468.75	712.11	12.42 o
-1	0	4	141.85	147.58	5.17 o
0	0	4	9248.00	9914.83	245.96 o
1	0	4	372.06	347.42	17.05 o
2	0	4	119.17	118.84	2.26 o
3	0	4	791.17	835.05	28.41 o
4	0	4	390.87	416.58	14.29 o
5	0	4	6.75	9.64	1.89 o
-6	1	4	543.62	521.62	17.91 o
-5	1	4	8.34	8.09	1.38 o
-4	1	4	318.68	302.34	7.66 o
-3	1	4	863.32	777.83	19.27 o
-2	1	4	9.78	5.69	0.73 o
-1	1	4	2426.76	2340.09	57.57 o
0	1	4	779.48	613.39	12.63 o
1	1	4	676.21	707.19	14.49 o
2	1	4	2112.11	2049.97	50.52 o
3	1	4	195.33	192.88	7.75 o
4	1	4	132.28	134.32	4.99 o
5	1	4	318.01	296.03	10.50 o
-6	2	4	1.85	3.96	1.55 o
-5	2	4	209.18	217.28	5.65 o
-4	2	4	2124.08	2085.30	51.42 o
-3	2	4	3.10	4.31	0.85 o
-2	2	4	2145.40	2173.61	53.55 o
-1	2	4	3425.97	3616.01	73.95 o
0	2	4	345.02	309.07	6.63 o
1	2	4	5306.75	4858.65	99.25 o
2	2	4	1465.05	1446.27	35.68 o
3	2	4	169.98	159.96	4.26 o
4	2	4	790.50	771.50	26.35 o
5	2	4	241.36	214.57	7.92 o
-6	3	4	458.65	434.66	14.98 o
-5	3	4	10.21	12.00	1.10 o
-4	3	4	337.24	341.62	8.57 o
-3	3	4	7.23	8.02	0.85 o
-2	3	4	3783.23	4046.36	82.72 o
-1	3	4	4.01	5.54	0.71 o
0	3	4	2119.55	2051.89	44.53 o
1	3	4	3280.91	3039.47	62.21 o
2	3	4	17.86	16.52	1.12 o
3	3	4	361.50	352.17	13.09 o
4	3	4	606.31	601.07	30.83 o
-6	4	4	183.49	181.34	4.87 o
-5	4	4	677.11	640.81	15.93 o

-4	4	4	62.92	59.36	1.88 o
-3	4	4	1723.99	1787.49	44.13 o
-2	4	4	787.24	790.22	20.45 o
-1	4	4	259.64	256.99	6.82 o
0	4	4	248.79	218.29	5.84 o
1	4	4	10.80	19.37	4.99 o
2	4	4	1.63	1.38	1.38 o
3	4	4	1209.38	1137.27	41.33 o
-5	5	4	1.61	2.41	1.55 o
-4	5	4	1056.57	1053.24	38.06 o
-3	5	4	491.19	524.65	13.75 o
-2	5	4	616.51	650.41	16.92 o
-1	5	4	194.03	179.15	4.92 o
0	5	4	300.71	291.43	7.79 o
1	5	4	4.47	6.62	2.58 o
3	5	4	126.04	118.14	5.17 o
-5	6	4	981.08	960.93	34.96 o
-4	6	4	123.27	142.72	4.19 o
-3	6	4	74.57	83.52	2.65 o
-2	6	4	352.94	348.98	9.64 o
-1	6	4	64.28	76.62	2.52 o
0	6	4	83.25	83.87	2.72 o
1	6	4	25.96	31.86	2.07 o
2	6	4	34.07	34.96	2.41 o
-4	7	4	64.70	68.45	2.57 o
-3	7	4	11.24	13.51	1.46 o
-2	7	4	112.05	124.52	3.70 o
-1	7	4	102.74	93.41	3.02 o
0	7	4	1018.53	1046.33	32.38 o
-2	-8	5	346.71	364.59	17.22 o
3	-8	5	508.09	476.85	16.19 o
4	-8	5	8.99	8.61	1.22 o
-3	-7	5	74.56	82.27	2.49 o
-2	-7	5	23.43	27.53	1.15 o
-1	-7	5	244.75	271.75	9.99 o
2	-7	5	515.41	466.52	16.88 o
3	-7	5	123.76	117.66	3.16 o
4	-7	5	109.04	107.50	3.04 o
-4	-6	5	4.03	5.07	1.40 o
-3	-6	5	17.02	17.99	1.27 o
-2	-6	5	875.86	935.44	23.10 o
-1	-6	5	8.05	6.68	0.78 o
2	-6	5	131.29	109.74	2.91 o
3	-6	5	130.02	130.31	4.22 o
4	-6	5	922.55	939.67	23.28 o
5	-6	5	97.53	114.52	3.35 o
-4	-5	5	2.95	0.63	1.32 o
-3	-5	5	4.48	3.27	1.15 o
-2	-5	5	717.82	822.96	20.36 o

-1	-5	5	23.51	33.42	1.10 o
0	-5	5	1307.34	1426.24	51.32 o
1	-5	5	214.33	185.48	4.91 o
2	-5	5	12.77	18.36	1.03 o
3	-5	5	1015.53	932.07	23.03 o
4	-5	5	264.68	276.90	7.05 o
5	-5	5	106.37	110.93	4.65 o
-5	-4	5	9.17	7.63	1.37 o
-4	-4	5	5.02	5.28	1.06 o
-3	-4	5	3918.29	4168.04	82.77 o
-2	-4	5	23.57	23.77	0.86 o
-1	-4	5	2866.11	3219.21	63.97 o
0	-4	5	1242.74	1224.78	32.56 o
1	-4	5	26.68	30.38	1.28 o
2	-4	5	1041.45	946.24	23.51 o
3	-4	5	966.29	940.06	18.80 o
4	-4	5	29.13	29.64	1.71 o
5	-4	5	770.86	815.22	16.35 o
-5	-3	5	318.59	326.11	6.82 o
-4	-3	5	232.21	238.03	4.97 o
-3	-3	5	253.82	274.59	5.59 o
-2	-3	5	1001.14	1104.67	22.04 o
-1	-3	5	3147.83	3611.69	71.78 o
0	-3	5	30.88	48.35	2.04 o
1	-3	5	2729.69	2691.66	53.51 o
2	-3	5	2907.23	2849.01	56.56 o
3	-3	5	7.70	5.70	0.75 o
4	-3	5	116.01	130.98	3.64 o
5	-3	5	369.01	376.45	13.09 o
-6	-2	5	86.03	90.75	3.96 o
-5	-2	5	348.13	341.49	11.88 o
-4	-2	5	198.05	195.62	4.14 o
-3	-2	5	1898.87	2031.84	40.44 o
-2	-2	5	661.66	576.48	11.55 o
-1	-2	5	407.30	464.79	9.33 o
0	-2	5	591.09	529.72	10.56 o
1	-2	5	1350.65	1225.50	39.56 o
2	-2	5	12.90	22.88	4.31 o
3	-2	5	353.79	321.88	6.55 o
4	-2	5	1124.73	1206.85	40.99 o
5	-2	5	361.48	370.25	12.92 o
-6	-1	5	3447.64	3099.43	104.70 o
-5	-1	5	163.54	157.40	5.86 o
-4	-1	5	411.18	400.96	8.14 o
-3	-1	5	339.75	323.38	9.69 o
-2	-1	5	115.41	99.21	2.12 o
-1	-1	5	210.70	185.61	3.81 o
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0	-3	8	122.79	162.79	4.61	o

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 2  -3   8       4.08       5.78      1.55 o
 3  -3   8     416.79     393.40      8.04 o
 4  -3   8     306.24     321.17     11.19 o
-5  -2   8       0.93       0.69      1.89 o
-4  -2   8     527.51     506.81     17.39 o
-3  -2   8     596.41     567.27     11.45 o
-2  -2   8       3.34       4.68      0.82 o
-1  -2   8     515.92     516.46     10.39 o
 0  -2   8       6.17       9.47      1.01 o
 1  -2   8       2.85       4.61      0.88 o
 2  -2   8     260.45     250.84      5.23 o
 3  -2   8     361.87     381.44     13.26 o
 4  -2   8      17.84      15.67      1.72 o
-5  -1   8     204.71     192.01      7.06 o
-4  -1   8    1105.88    1112.82     37.71 o
-3  -1   8      58.76      61.31      2.76 o
-2  -1   8     553.43     578.99     11.65 o
-1  -1   8    3687.28    3637.48     72.31 o
 0  -1   8     874.23     924.58     18.50 o
 1  -1   8    2308.39    2346.88     79.22 o
 2  -1   8     519.92     528.17     18.08 o
 3  -1   8       0.19       0.69      1.55 o
 4  -1   8     690.46     690.56     23.59 o
-6   0   8     319.88     282.42     10.16 o
-5   0   8     446.42     439.48     15.33 o
-4   0   8     447.59     427.60     14.81 o
-3   0   8       1.98       3.96      1.55 o
-2   0   8    4608.07    4597.32    154.99 o
-1   0   8     365.02     357.94      9.11 o
 0   0   8    4199.78    4262.88    143.80 o
 1   0   8    2528.20    2473.62     83.52 o
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 3   0   8     218.82     211.82      7.58 o
 4   0   8     504.13     509.05     17.57 o
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-5   1   8     354.49     334.09     11.71 o
-4   1   8     361.07     353.20     12.23 o
-3   1   8     309.54     320.65     11.19 o
-2   1   8     710.11     694.86     14.39 o
-1   1   8     176.01     167.52      3.67 o
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-5   2   8       0.49       2.41      1.89 o
-4   2   8     350.49     347.35     12.05 o
-3   2   8     494.86     483.42     12.16 o
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-1	0	12	13.81	21.01	2.41 o
0	0	12	153.13	143.28	5.68 o
1	0	12	12.13	21.18	2.41 o
-3	1	12	696.03	675.06	23.25 o
-2	1	12	125.79	128.64	5.17 o
-1	1	12	0.22	1.03	2.24 o
0	1	12	439.84	484.43	16.88 o
-1	-5	13	117.51	120.82	3.71 o
0	-5	13	0.45	0.61	1.76 o
-2	-4	13	757.05	780.11	26.86 o
-1	-4	13	170.62	194.77	7.41 o
0	-4	13	490.69	572.94	19.80 o
-2	-3	13	8.58	8.27	2.58 o
-1	-3	13	341.27	393.67	13.78 o
0	-3	13	298.01	377.14	13.43 o
1	-3	13	36.18	39.09	2.93 o
-3	-2	13	17.56	21.35	2.76 o
-2	-2	13	339.19	336.33	12.05 o
-1	-2	13	131.70	148.10	5.86 o
0	-2	13	20.32	21.18	2.58 o
-2	-1	13	163.00	162.39	6.37 o
-1	-1	13	46.53	50.29	3.10 o
0	-1	13	106.65	124.16	5.17 o
-2	0	13	44.76	48.39	3.10 o
-1	0	13	132.32	143.11	5.68 o

===END of fcf